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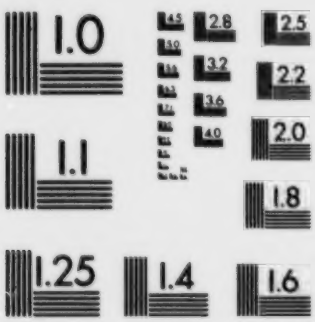


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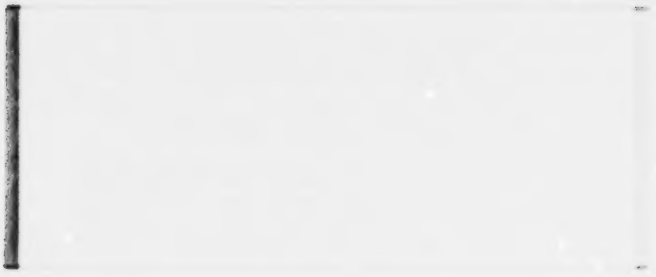

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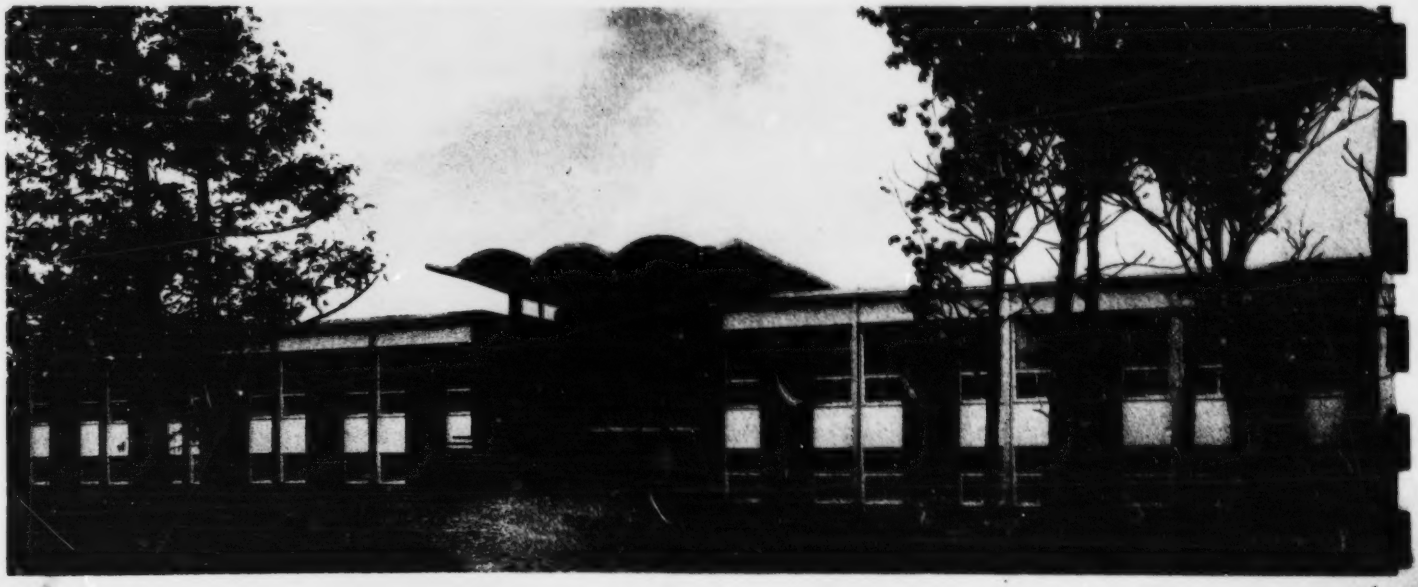

# MODIFICATION OF INTEGRATED OIL SHALE RETORT SYSTEM
(50/50 Cost Shared Energy Conversion Program)

Final Report
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ABSTRACT

The combustor retort system was redesigned and rebuilt. The pilot plant consists of a circulating fluidized bed combustor of 13 cm internal diameter, primary cyclone and standpipe, J-valve, heating and cooling system and baghouse. The retort has an inside diameter of 25.4 cm and is 2.9 m high with solids feeding and withdrawal system, equipment for product oil recovery, a gas recycling net work and gas sampling system. A heat exchanger was built specially for using the combustor heat for the retorting.

Retorting with the indirect method by heating of the circulating gas gave good oil yields. There were no difficulties with combustor performance when fired only on coal. Firing the circulating fluidized bed with spent shale of large size with the coal resulted in clinkering. Hence the fully integrated system with the combustor burning coal and spent shale to produce heat for the retorting of raw shale and the collection of a liquid petroleum product was limited.

More work is advised in order to make the final break through of continuous controlled operation of the integrated system. The results will be relevant to the CANMET/NBEPC circulating fluidized bed demonstration project at Chatham.

## INTRODUCTION

The integrated combustion/retorting system for coal and oil shale was devised for the co-utilization of high sulphur coal from eastern Canada and the oil shale from New Brunswick. Previously it had been shown that the prospects for economic development of the oil shales were best when done in conjunction with coal use*.

In this process liquid petroleum is produced, excess heat can be used for steam raising and the sulphur emissions are controlled. In this integrated process the oil shale is retorted to skim off the volatile oil and gases. The spent shale, containing residual kerogen is combusted with high sulphur coal in a circulating fluidized bed combustor. The magnesium and calcium content of the shale make it an effective sulphur captor. In addition, it has a calorific content of 930-1396 KJ/kg which is utilized during combustion. The heat required for retorting is supplied by the flue gas from the combustor either by indirectly heating the recycle retort gas or by direct introduction into the retort.

In the earlier work RPC designed, constructed and operated a 0.6 tpd pilot scale retort in both the direct and indirect heating modes for extended periods of time lasting up to 2 weeks of continuous operation.** In the direct mode heat was supplied by burning the shale at the base of the moving column in the

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* RPC report MDP/82/31. Design Concepts and Preliminary Economics for Coal/Oil Shale Combustion Retort Systems.

** RPC report AS/85/51. Demonstration of an Integrated Oil Shale Retort System. December 1984.

retort. Indirect heating involved injecting hot gases into the shale in the retort. Oil recoveries were 80 to 90 per cent of Fischer assay. The product shale oil is of low density and relatively low in sulphur, nitrogen and oxygen which makes it an excellent refinery feedstock. During the combustion of the spent shale to utilize the 30 per cent energy remaining, sulphur capture was approximately 90 per cent at a Ca/S ratio of 3.0. Full integration between the combustion and the retort was not achieved because of the design of both the combustor and retort. Further funding was sought to continue this work.

In July 1985 a contract was issued under the 50/50 Cost Shared Energy Conversion Program for the continuation of that project. This report is an account of the work undertaken in fulfillment of that contract*. The work statement is given in Appendix A. In summary there were five tasks.

1. Modification of the Combustion System
2. Sample Preparation
3. Retorting with Direct Introduction of Combustion Gases
4. Indirect Heating of Recirculating Gas from Retorting
5. Report Preparation

It was felt that if RPC is to continue to lead oil shale and high sulfur coal development work in Eastern Canada, units of advanced design were essential. With this in mind, RPC replaced or redesigned all the old equipment which were not originally designed as part of the integrated system. The total cost of the

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* Modification of Integrated Oil Shale Retort System (50/50 Cost Shared Energy Conversion Program) DSS contract 06SQ23440-5-9028

system is approximately $490,000. Although Federal and Provincial Govt. contributed a total sum of approximately $160,000 through the 50/50 cost shared programs, RPC's contribution for the construction of the system was over $330,000. It is hoped that these large expenditures will be recouped in subsequent contract work.

Since commissioning, at the end of 1985, the circulating fluidized bed combustor has been used for contract work on combustion and waste production with coal from Nova Scotia, New Brunswick, Pennsylvania and Saskatchewan with various limestones.* This work was on too small a scale to reimburse RPC but it did show the excellent performance possible with the circulating fluidized bed combustion.

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* RPC confidential report AS/87/03. "Production of Circulating Fluidized Bed Combustion Wastes for Characterizations."

TASK 1 MODIFICATION OF THE COMBUSTION SYSTEM

Activities were to have included dismantling and reconstructing the 6 inch fluidized bed combustor and to installing a heat exchanger to utilize the heat of combustion to raise the temperature of the recirculating gases from retorting.

Modification of the 6 inch fluidized bed combustor proved impractical. In another contract, cost shared between the Atlantic Research Laboratories of NRC and Provincial Holdings Ltd. of New Brunswick in Fredericton, construction had commenced on a refractory-lined circulating fluidized bed combustor* (Appendix B).

It has been reported** before that the old retort had leaks at different locations which were difficult to repair. These leaks were responsible for undesirable air infusion into the system. A new cylindrical retort was designed and commissioned to overcome these problems. Along with the retort, a new knock out pot was designed and installed to replace the existing hot cyclone. The performance of the old absorption column and the electrostatic precipitator which were originally designed for some other purpose were not entirely satisfactory. These two units were replaced with units of better design. Also a specially designed heat exchanger was fabricated and installed for this particular project. All in all, an entirely new integrated system was designed, installed and commissioned.

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* RPC Report AS/86/16. Pyrolysis of Eastern Canadian Coals and Oil Shales in a Circulating Fluidized Bed Reactor. Phase 1 - Construction of Unit.

** RPC Report AS/85/51, "Demonstration of an Integrated Oil Shale Retort System", Final Report, December 14, 1984.

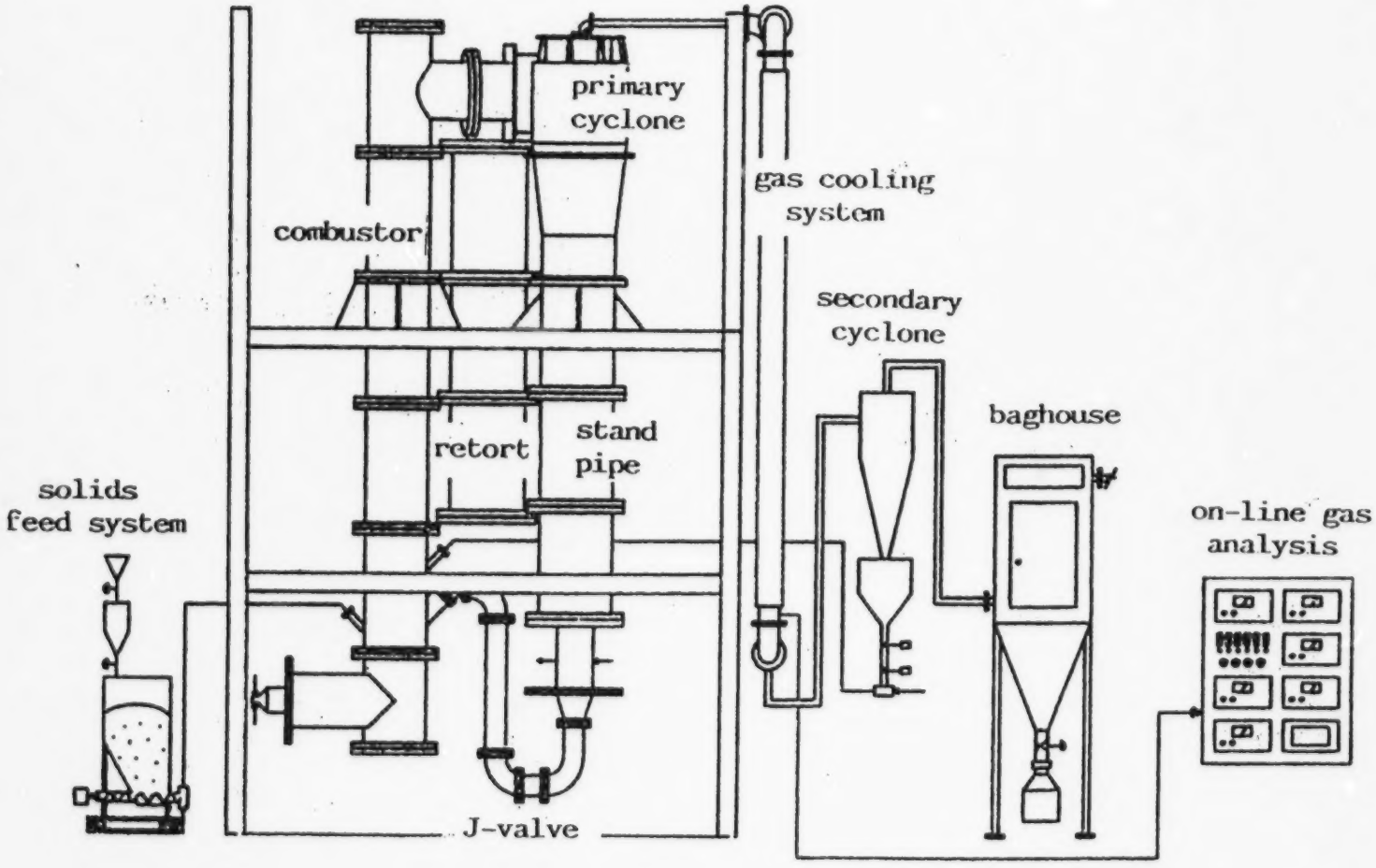


FIGURE 1 CIRCULATING FLUIDIZED BED REACTOR

## 1.1 The Circulating Fluidized Bed Combustor

The circulating fluidized bed consists of a combustion chamber, a primary cyclone and a stand pipe, a gas cooling system, a secondary cyclone and a baghouse. Solids are fed to the system by gravity or pneumatically (Figure 1). A brief description of the major components is given below.

### 1.1.1 The Combustor

The combustor is cylindrical in shape with a total height of 5.8 m (19') and it has an inside and outside diameter of 12.7 cm (5") and 50.8 cm (20") respectively. The wall consists of three layers of material: an inner layer made up of high temperature refractory, a middle layer of castable insulating refractory, and a carbon steel shell. The thickness of the high temperature refractory and insulating are 5 cm and 13 cm, respectively. A 0.5. MBtu propane burner is used for start-up purposes.

### 1.1.2 The Stand Pipe

The stand pipe is used for storage and recirculation of solids received from the primary cyclone. It is 2.7 m (9') high and has an inside and outside diameter of 22.8 cm (9") and 61 cm (24") respectively. The stand pipe is also refractory lined and it is also equipped with discharge and sampling ports.

A cooling section is installed at the bottom of the stand pipe. This is used to extract the extra heat generated in the combustor so that both bed temperature and air/fuel ratio can be set and maintained at the appropriate levels.

### 1.1.3 Cyclones

The primary cyclone which is 30 cm (12") in diameter, and is refractory lined in a similar manner to the reactor and the standpipe. The secondary cyclone is made of stainless steel and is 25 cm (10") in diameter. All the solids collected by the secondary cyclone are recycled pneumatically to the combustor.

### 1.1.4 J-Valve

Circulation of solids from the standpipe to the reactor is achieved by the non-mechanical J-valve. Air is supplied at two points - one at the base of the standpipe, and the other is at the base of the vertical section of the J-valve. The J-valve is made of 2", 40 Schedule SS pipe.

### 1.1.5 Heating and Gas Cooling System

Hot flue gases from the combustor was fed into a specially designed heat exchanger as described below. (See Figures 2 and 3). The gases were then cooled further in water jacketed pipes which simulate the convective section of a boiler. The cooling is carried out in a water cooled one pass heat exchanger which is made of two concentric pipes of a nominal diameter of 15 cm (6") (Schedule 40) and 20 cm (8") (Schedule 60), respectively. The height of the exchanger is 5.2 m (17").

### 1.1.6 Baghouse

The flue gases from the cooling section are fed through the secondary cyclone into the baghouse. The latter is a continuous duty self cleaning dust collector which consist of 16 high temperature bags capable of handling 200 SCFM.

### 1.1.7 Solid Feeding to the Combustor

The solids, discharged by a screw feeder from a storage bin, are entrained by air and fed to the combustor. The storage bin is placed on a load cell to provide an account of the solid feed rate.

## 1.2 The Retorting System

The system consists of a cylindrical retort, solid feeding and withdrawal equipment, product oil recovery system, and gas recycling network. Each component of the system is described below:

### 1.2.1 The Retort:

The retort has been designed to minimize undersirable air infusion and to withstand slightly elevated pressure (5PSi). Our previous experience showed the spent shale discharge screwfeeder tended to seize due to the generation of spent shale fines, thus disrupting the operation. A gate valve has been installed at the bottom of the retort so that the discharge screwfeeder can be taken down for maintenance without emptying the retort.

The cylindrical retort has an inside and outside diameter of 25.4 cm and 61 cm respectively and a total height of 2.9 m. Like the combustor, the wall consists of three layers of material: an inner layer of castable high temperature refractory, a middle layer of castable insulating refractory and a carbon steel shell. The thickness of the high temperature and insulating refractory are 11.7 cm and 5.1 cm respectively.

The cleaned retort off-gases are recycled to the retort as bottom and middle recycle gas. The middle recycle gas, after spliting into two streams, is introduced at a height of 0.8 and 1.25 m measured from the bottom of the retort.

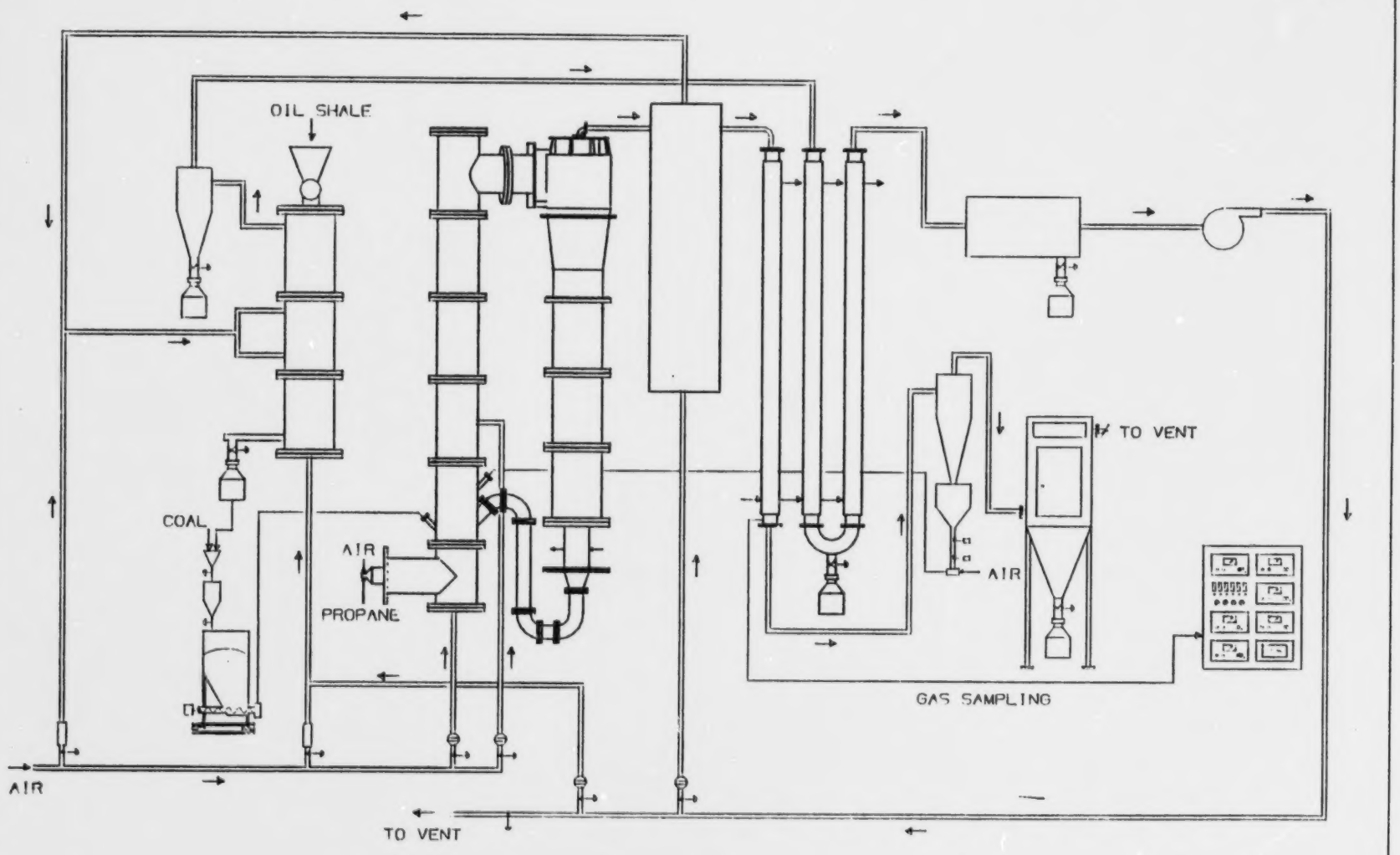


FIGURE 2 : SCHEMATIC OF RPC INTEGRATED SYSTEM

The oil, mist-ladden, off gases are withdrawn near the top of the retort through a side pipe equipped with a baffled cup to minimize dust entrainment which can cause line pluging.

### 1.2.2 Solid Feeding and Withdrawal System

Oil shale is fed into the retort by gravity from an overhead hopper through a rotary valve. A predetermined amount of oil shale is loaded into the bin every hour and a timer is used to control the ON/OFF cycles of the rotary valve in order to maintain a steady supply of fresh shale.

Spent shale is discharged from a bin attached to the bottom of the retort through a variable-speed screwfeeder into a receiving pot. The discharge rate is set so as to maintain a steady shale bed height in the retort. Bed level is measured periodically with a graduated rod.

### 1.2.3 Product Oil Recovery System

Off-gas containing oil mist from the retort undergoes three stages of separation for product oil recovery. These take place in:

(a) a hot knock-out pot for removing heavy oils and entrained fine particles.

(b) two condenser columns packed wtih rasching rings, with an internal column diameter of 0.15 m and an effective height of 4.7 m.

(c) an electrostatic precipitator (0.55m wide x 1.52m long x 0.13m thick) with five sets of capacitor cells and ionizer. The operating voltages on the cells and ionizers are 5 KV (2mA) and 10 KV (20 mA) respectively. It was found necessary to reheat the gas above the dewpoint and to operate the electrostatic precipitator in the temperature range of 65-70°C in order to prevent undersirable discharge of cell and ionizer voltages due to water condensation.

### 1.2.4 Gas Recycling Network

The cleaned off-gases from the oil recovery system is metered and recycled back to the retort. A portion can be withdrawn as a product gas. The middle recycle can be preheated in the heat exchanger prior to injection to the two levels of gas feed points in the retort. At each level, recycle gas is distributed by 2 cm diameter SS 316 pipes imbedded in the shale which are placed at 90° with respect to each other. Each pipe contains 10 holes of 0.24 cm diameter.

### 1.2.5 Gas Sampling System

On-line gas analyzers (hydrocarbon, CO, $CO_2$, $O_2$, $NO_x$, $SO_2$) are used to monitor the composition of the retort and combustion gases. Retort gas samples are withdrawn from the recycle line and conditioned via a series of filters (knock-out pot, impingers, heated by-pass filter, cloth filter, and dryers) prior to entering the analyzers. Combustion flue gas samples are withdrawn through a heated line and cleaned by a series of filters. One stream, either combustion flue gas or retort off gas, is continuously monitored, while the other is analyzed as necessary.In addition, tubes of retort gas sample are also collected for further analysis of $H_2$ and $N_2$ content and hydrocarbon distribution using gas chromatography.

## 1.3 The Heat Exchanger

The heat exchanger, which has been used for the heating of the middle recycle gas with the hot flue gases from the combustor, has been designed to meet the specifications given in Table 1. A summary of the detailed design is given in Table 2. Figure 3 shows the schematic of the heat exchanger which was fabricated in RPC's workshop.

Table 1

Heat Exchanger Specifications

| * Component | Gas Composition | |
|---|---|---|
| | Retort Gas | Flue Gas |
| $CO_2$ | 6.0 | 16.0 |
| CO | 4.2 | - |
| $H_2$ | 4.3 | - |
| $O_2$ | 1.3 | 2.0 |
| $H_2O$ | - | 6.6 |
| $N_2$ | 79.4 | 74.6 |
| $SO_2$ | - | 0.8 |
| $CH_4$ | 1.1 | - |

* Amount of flue gas: 80 - 140 kg/hr
* Amount of recycle gas: 20-40 kg/hr
* Temperature of flue gas: 800°C
* Dust loading of flue gas: 5-15 $g/m^3$, size 10 m
* Inlet and desired outlet temperature of recycle gas: 25° and 700°C
* Pressure: atmospheric

TABLE 2
SUMMARY OF DESIGN DETAILS

Overall Heat Transfer Coefficient - 11.35 $w/m^2C$ (2.0 BTU/hr-$ft^2F$)

Heat Transfer Surface Area - 4.2 $m^2$ (45 $ft^2$)

Heat Exchanger configuration - counter-current
- "No tubes in the window" design

Tubes

O.D. of tubes - 25.4 mm (1.0")

Number of tubes - 11 (Eleven)

Arrangement - "Staggerred", triangular pitch pattern

Pitch of tubes - 38 mm (1.5 inches)

Number of tube rows
in the direction of gas flow -3 (three), see sketch attached

Average tube length = 4763 mm (15.63 ft)

Baffles

Standard drilled plate type. "TEMA" standards was used for hole tolerances. Tie rods could be used for positioning baffles.

Baffle spacing - 305 mm (12 inches) max.

Nozzles

All nozzles are 89 mm I.D. (3.5 inches) minimum.

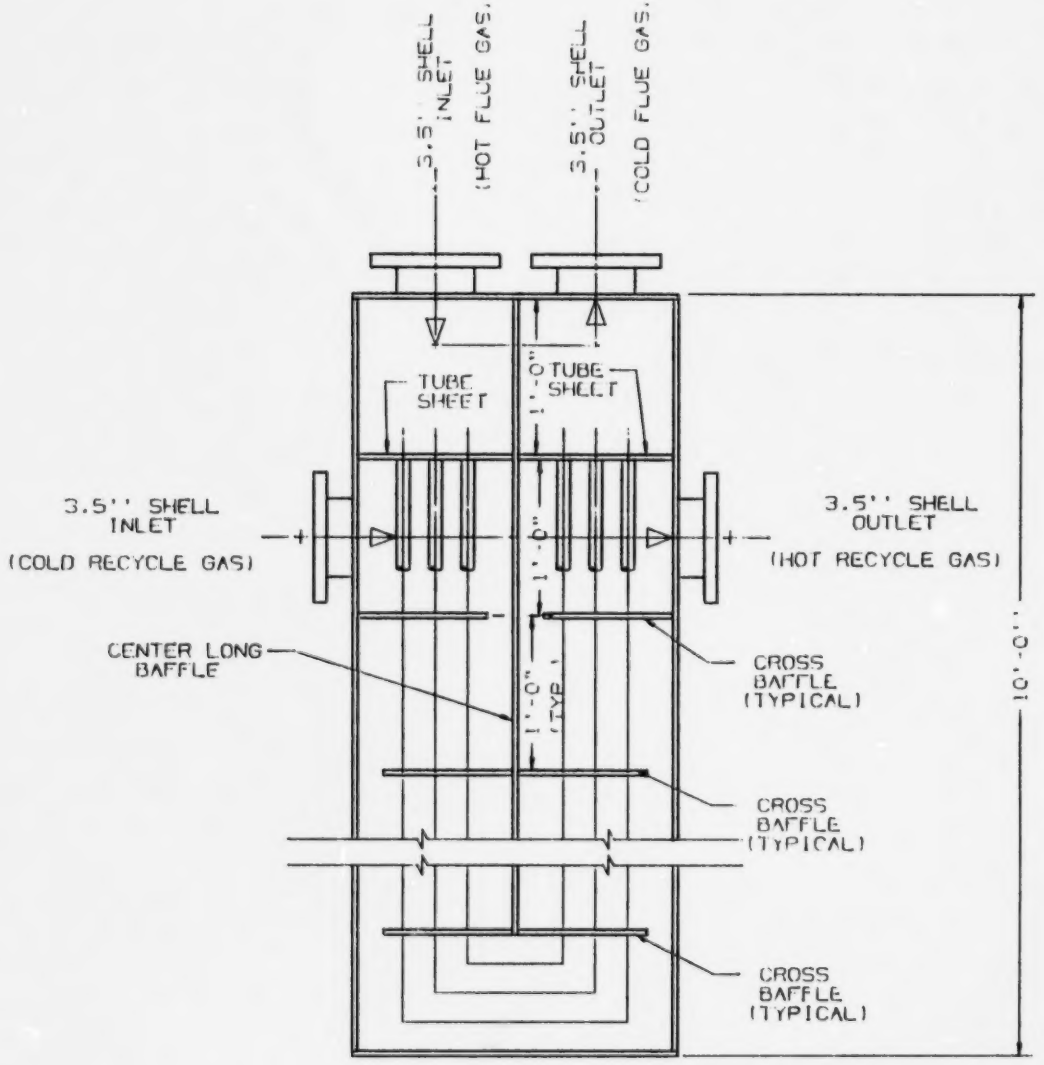


FIG 3 SCHEMATIC OF THE HEAT EXCHANGER FOR THE INTEGRATED SYSTEM

TASK 2 SAMPLE PREPARATION

Supplies of oil shale and coal from the previous retort work were insufficient for the present project and additional oil shale and coal had to be obtained. As received crushed oil shale was dried and screened through 5/8" and 1/8" to remove oversize and undersize. The as-received coal was dried, crushed and screened. The chemical analyses and the Fischer assay analysis of oil shale, coal and spent shale are given in Table 3.

TABLE 3
CHEMICAL ANALYSIS OF COAL AND SHALE

| | Coal* | Tests Shale 1 | Phase 1 Shale 2 | Present Tests Coal | Shale | (Phase 2) Spent Shale |
|---|---|---|---|---|---|---|
| **Proximate Analysis (wt%)** | | | | | | |
| Moisture | 0.81 | 0.68 | 0.43 | 0.62 | 1.76 | |
| Ash | 16.6 | 77.55 | 83.16 | 14.20 | 80.98 | |
| Volatile Matter | 33.8 | 21.77 | 16.41 | 35.60 | 15.50 | |
| **Ultimate Analysis (wt %)** | | | | | | |
| Carbon | 67.3 | 14.76 | 11.93 | 70.66 | 12.06 | 9.34 |
| Hydrogen | 4.63 | 1.86 | 1.48 | 4.76 | 2.01 | 1.24 |
| Nitrogen | 0.72 | 0.10 | 0.30 | 0.95 | 0.44 | 0.41 |
| Oxygen | 3.46 | 4.96 | 2.50 | 2.50 | 2.10 | 4.49 |
| Sulphur | 7.29 | 0.77 | 0.63 | 6.30 | 0.10 | 0.28 |
| Ash | 16.6 | 77.55 | 83.16 | 14.20 | 80.98 | 84.24 |
| **Ash Analysis (wt%)** | | | | | | |
| $SiO_2$ | 28.8 | 57.82 | 63.71 | | 61.94 | - |
| $Al_2O_3$ | 19.3 | 15.62 | 17.14 | | 16.16 | - |
| $Fe_2O_3$ | 48.7 | 7.86 | 4.45 | | 6.29 | - |
| CaO | 0.37 | 10.18 | 5.70 | | 3.74 | 9.02 |
| MgO | 0.26 | 4.09 | 2.94 | | 2.24 | 2.17 |
| $Na_2O$ | 0.41 | 2.11 | 3.88 | | 1.90 | - |
| $K_2O$ | 0.60 | 2.32 | 2.18 | | 3.20 | - |
| S(as $SO_3$) | - | | | | | |
| **Gross Heating Value** | | | | | | |
| (Btu/lb) | 12583 | 2005 | 1700 | 12066 | 2056 | |
| MJ/Kg | 29.13 | 4.64 | 3.94 | 29.79 | 4.76 | |
| **Fischer Assay (wt%)** | | | | | | |
| Oil Yield | | 7.76 | 5.91 | | 7.2 | |
| $H_2O$ yield | | 1.80 | 1.70 | | 4.0 | |
| Gas lost | | 1.14 | 2.39 | | 16.5 | |
| Oil yield (Imp Gal/ton) | | 21.0 | 15.5 | | 0.868 | |
| Density (g/cc at 15.5 °C) | | 0.7391 | 0.7592 | | | |

* Average of two samples

## TASK 3 RETORTING WITH DIRECT INTRODUCTION OF COMBUSTION GASES

In this task part of the hot flue gases from the combustor was to be fed directly into the retort, thus supplying the heat requirement. Previous experience, has shown that this type of operation leads to difficulties because the dust in the flue gas plugs the distribution pipes inside the retort. The consequence is back-pressure on the combustor and operational difficulties such as interruption in solid feeding and possible bursting of combustor and cyclone gaskets.

For the reasons given above it was decided to carry out Task 4, first then proceed with this task. However due to time and budget constraints, direct introduction of flue gases have not been attempted as yet.

# TASK 4 INDIRECT HEATING OF CIRCULATING GAS

## 4.1 Operational Procedures

Preheating of the retort and the circulating fluidized bed combustor was initiated simultaneously. Immediately after the start-up the units were operated independently. The retort was in the direct operating mode i.e. the heat requirements of the retort were generated inside the retort itself. The combustor was on coal feed.

### 4.1.1 Retort Start-Up

Retort preheating was started with a propane burner placed through a side port located close to the bottome of the retort. When the temperature at the top of the retort was approximately 900$^{o}$C, the burner was removed and the side port was sealed. The retort was immediately filled with oil shale and operated in the direct retorting mode. Recycling of the cleaned product gases was also initiated immediately. The product gases were passed through the cleaning train but not the electrostatic precipitator because of the high oxygen concentration. Total preheating time was approximately four hours.

### 4.1.2 Combustor Start-Up

The standpipe was filled with 200 kg of sand prior to preheating of the combustor with a 0.5 million Btu/hr propane burner. When the temperature of the combustor was over 700$^{o}$C, recycling of the sand from the standpipe was initiated. The standpipe was preheated by the hot recycling sand. Coal feed was initiated when the standpipe temperature was over 600$^{o}$C. Preheating of the combustor system took over twelve hours. Steady combustor operation was achieved in approximately 16 hours. Combustor temperature was maintained by adjusting the coal feed rate and by controlling the heat removal rate by the cooling section.

### 4.1.3 Integrated System

The combustor and retort layout has been designed such that the spent shale can be fed directly into the combustor. This is to be achieved using a screw conveyor at the bottom of the retort which discharges into an included ($45^{o}$) side port through the combustor wall. This concept however was not implemented because of the large particle size of the spent shale which is unsuitable for the operation of a circulating fluidized bed. Instead an intermediate step was necessary to process the spent shale i.e. (particle size reduction) before it can be fed into the combustor. It is important to note that this limitation may no longer exist with scale up or the use of a bubbling bed.

In this project integration of both systems was carried out when sufficient amount of spent shale was generated. The spent shale was screened to a particle size of $-\frac{1}{4}$" and then blended with the coal at a ratio of 1:1. The recycled retort gases were directed through the heat exchanger for preheating prior to reintroduction into the retort.

### 4.2 System Performance

The system was operated in the integrated mode on three separate occasions. In all cases the run was terminated because of clinker formation inside the combustor. This is because spent shale particles are less susceptible to attrition as compared to coal particles (less burning). The accumulation of large amounts of spent shale particles inside the combustor, lead to de-fluidization and clinker formation. Operational and equipment changes introduced in an attempt to solve the clinkering problem by removal of large particles included:

i Installation of a bed drain at the side of the combustor , 45" above the distributor plate.

ii Installation of a bottom drain, which can be adjusted to alter the bed height. Bed height attempted was 28".

The longest recorded run of the integrated system was 17 hours before clinkering occurred inside the combustor.

Had more work been possible under the contract fine sized spent shale would been used. The clinkering problem would probably not occur under those circumstances.

4.2.1 Retorting Performance

The recorded data for the last 13 hours of the operation of the integrated system are reported and analysed here. The retort operating conditions were as follows:

| | |
|---|---|
| Shale feed rate: | 16.9 kg/hr |
| Average bed height: | 2 m |
| Shale residence time: | 6.7 hr |
| Total recycle gas/100 kg of shale: | 228.2 kg |
| Recycle gas to the middle: | 79.4% |
| Recycle gas to the bottom: | 20.6% |
| Temperature of middle recycle before entering the retort: | 553-564°C |
| Air feed to the bottom per 100 kg of shale | 17 kg |
| Estimated oil recovery: | 95-109% of Fisher assay |

The overall mass balance for the thirteen hour period is shown in the Figure 4. The oil recovery is over 95% of the Fisher assay oil. The temperature distribution is qualitatively similar to the previous indirect retorting experiments. However, maximum temperature recorded was only 510°C in the retort which is lower than in the previous indirect retorting experiments. Because of the higher bed height and low feed rate, the shale residence time is higher than the previous retorting experiments.

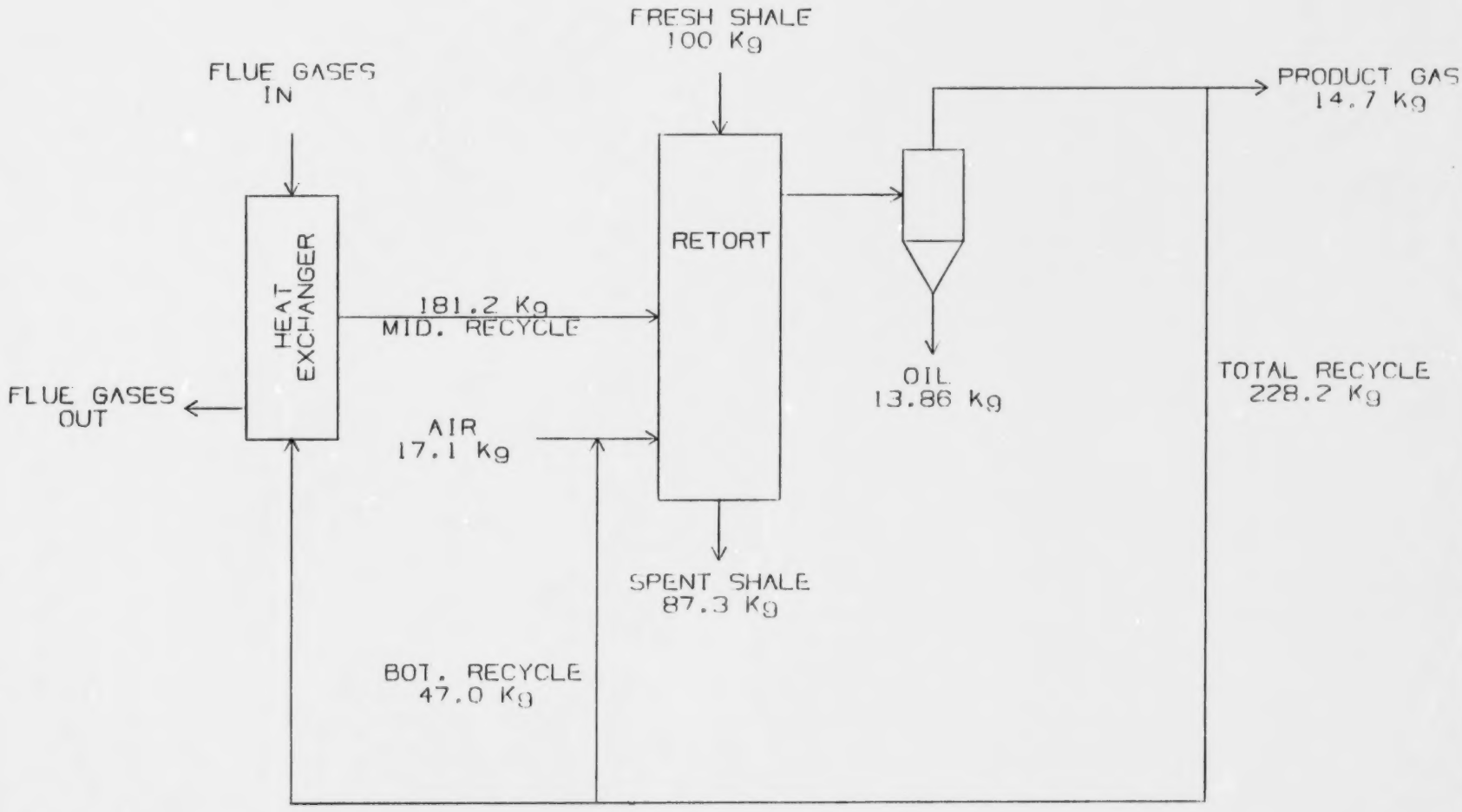


FIGURE 4 : SUMMARY OF MASS BALANCE

Although total recycle gas is in the same range as other retorting experiments, similar distribution did not produce the best oil recovery. The concentration of carbon monoxide and carbon dioxide in the product was 2.7% and 7.3% respectively. The oxygen concentration in the flue gas could not be detected suggesting that the unwanted oxygen infusion to the retort was minimal.

Elemental analysis of shale oil from the present program and previous indirect retorting experiments are shown in Table 4. The Present oil is somewhat richer in hydrogen as reflected in the H/C ratio. Nitrogen content of the present oil is lower than the oil produced in the previous program, while sulfur and oxygen contents are higher.

Table 4

Elemental Analysis of Shale Oil

| Components | Weight % Present Program | Previous Indirect Expt (Shale 2 Run 27) | (Shale 1 Run 13) |
|---|---|---|---|
| Carbon | 84.96 | 86.46 | 86.51 |
| Hydrogen | 11.94 | 11.80 | 11.95 |
| Nitrogen | 0.60 | 1.16 | 0.79 |
| Sulfur | 0.56 | 0.28 | 0.47 |
| Oxygen | 1.94 | 0.30 | 0.28 |
| H/C | 1.69 | 1.64 | 1.66 |

### 4.2.2 Heat Exchanger Performance

The heat exchanger was designed to heat the middle recycle gas to 700°C. The maximum temperature recorded at the outlet of heat exchanger was 520°C during the experiment even though the gas recycle rate did not exceed the designed flow rates. The heat exchanger was opened at the conclusion of the program. Four heat exchanger tubes out of eleven were plugged, which means that the effective heat transfer area was reduced by 37%. It is believed, however, that this plugging might have accured during the debugging of the combustor prior to the start of this particular test program. i.e. in normal operation this may no longer be a problem. Should the problem persist however, a secondary cyclone can be installed before the gases are fed into the heat exchanger.

In this test and in order to compensate for the lower temperature at the exit of the heat exchanger, the pipe leading from the heat exchanger outlet to the retort inlet was traced with electrical coils. Maximum retort inlet temperature recorded was 564°C.

### 4.2.3 Combustor Performance

The operating parameters of combustor are as follows:

| | |
|---|---|
| Solid Feed Rate: | 13.1 kg/hr |
| Coal: Spent Shale: | 1:1 |
| Air Feed Rate: | 117.8 kg/hr |
| Ca/S Ratio: | 0.9 - 1.2 |
| Temperature at Combustor Exit: | 854° + 5°C |
| Estimated Excess Air: | 56% |
| $SO_2$ in the Flue Gas: | 2500 ppm |
| Theoreticals $SO_2$ in the Flue Gas: | 3400 ppm |
| % S Capture | 26% |

Sulphur capture was less than expected because of low shale to coal ratio and high concentration of sand in the recycled solid. An extended run with high shale to coal ratio is expected to improve the sulfur capture. In another test program, combustion trials with high sulphur coal and limestone have achieved 97% sulphur capture at a Ca/S ratio 2.5:1.

## CONCLUSIONS AND RECOMMENDATIONS

The newly designed retort performed very well, the oil yield achieved was over 95% of the Fischer assay oil yield.

In the integrated mode, the system operated continuously for seventeen hours, after which clinker formed in the combustor due to the accumulation of large size shale particles. It is likely that the spent shale has to be reduced in size, then screened before it can be fed to the combustor.

This investigation indicated, that all the essential components needed for successful operation of an integrated system are available. However, some more debugging will be necessary which will require additional funds and time. Given that, the system can be made to operate reliably for extended period of time as an integrated system.

Further work on the integrated system is strongly recommended in order to demonstrate the system operatively and to optimize the process in terms of overall energy efficiency and product state.

# AUTHORIZATION

DEPARTMENT: APPLIED SCIENCE

PROJECT TITLE: MODIFICATION OF INTEGRATED OIL SHALE RETORT SYSTEM

PROJECT NUMBER: 05000218 PROPOSAL NUMBER

REPORT AS/87/05

Prepared By: P. Salib
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Reviewed By: P. Salib

Approved By:

Date: June 10 1986

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# APPENDIX A
# WORK STATEMENT

APPENDIX "A"

TITLE: MODIFICATION OF AN INTEGRATED OIL SHALE RETORT SYSTEM
(50/50 COST SHARED ENERGY CONVERSION PROGRAM)

The work of this phase of the contract will be performed in the following tasks:

Task 1 Modification of the Combustion System

The activities of this task will include:

- dismantle the existing combustor
- install the new refractory lined combustor
- make all necessary connections to the retort, feeding system and discharging system
- check for possible leaks
- install a heat exchanger to utilize the heat of combustion gases to heat up the recirculating gas from the retorting

Task 2 Sample Preparation

- use the oil shale sample #1 from the Phase I (the same particle size distribution)
- use the same sample of Minto coal as used in the Phase I

Task 3 Retorting with Direct Introduction of Combustion Gases

In this task the following steps will be taken:

- on the basis of experimental experience from the Phase I select the optimal conditions for retorting and subsequent co-combustion.
- under the selected conditions perform one experiment in the integrated mode; i.e., attain the steady-state while the combustion gases vented out. After the steady state is reached introduce the hot combustion gases into the retort.
- monitor carefully the changes of pressure, in case that leaks develop as the result of pressure build up abort the experiment.

APPENDIX "A"

Task 4 Indirect Heating of Recirculating Gas from Retorting

This task will include the following activities:

- estimate the heat requirements to maintain the temperature of recirculating gas from retorting at 700°C, via indirect heating using hot combustion gases.
- perform a separate combustion experiment to verify the heat balance calculations; experimentally determine the required temperature of combustion.
- perform a long continuous experiment (2 days) in the integrated mode; determine the yields of liquid products, change in the composition of gaseous products (recirculating gas), composition of combustion products; ensure an accurate measurement of temperature of recirculating gas at the exit from the retort, at the injection part of the retort and the temperature of combustion gases at the exit from the combustor and from the heat exchanger.
- at the end of the experiment open the heat exchanger and check for a possible accumulation of dust and abrasion.

Task 5 Report Preparation

The report will include:

- detailed description of all modifications.
- description of the operating procedures during the direct injection of hot combustion gas as well as using the external heating of recirculating gas by hot combustion gases.
- results of the analysis of recirculating and combustion gases, of raw and spent oil shale, of Minto coal and of liquid products.
- recommendations and conclusions.

The Go-No-Go decision will follow the completion of the Phase II. The Go decision is subject to a successful completion. Then, the main objective of the continuation of this work will be the long runs (up to seven days) to obtain the technical data for the scale up considerations.

# APPENDIX B
# DESCRIPTION OF UNIT CONSTRUCTION

# PYROLYSIS OF EASTERN CANADIAN COALS AND OIL SHALES IN A CIRCULATING FLUIDIZED BED REACTOR

Phase One-Construction of Unit

Final Report Prepared for

S.G. Whiteway, Scientific Authority
Atlantic Research Laboratory, NRC, Halifax

and

D.E. Gemmell
Provincial Holdings Ltd, Fredericton

Report No. AS/86/16
Job No. 50214
Date: August 16, 1985

P. Salib
Head, Fuel Technology
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Process Engineer
Fuel Technology Section

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## ABSTRACT

This report describes the design and construction of a circulating fluidized bed reactor which can be used to investigate the production of liquid petroleum production from coal and oil shale. It consists of a fast fluidized bed reactor, stand pipe, cyclones, gas and liquid cooling and condensing systems, solid and gas recycling network and a feeding system. Cold tests were performed to demonstrate that the design was suitable for effective circulation of the solids.

## 1.0 INTRODUCTION

The work described in this report was in fulfillment of DSS contract File No. 12SC31028-4-4962Y dated 26 November 1984, which had been issued in response to the RPC revised proposal AS/85/7-P of September 1984 "Pyrolysis of Eastern Canadian Coals and Oil Shales in a Circulating Fluidized Bed Reactor". That proposal had requested through the Atlantic Research Council (ARL) of NRC, Halifax a total of $161,670 for a two-part project. The first, dealing with design, construction and modification of the experimental facility and its commissioning, was budgeted at $84,000 to be cost-shared by Provincial Holdings Ltd, Fredericton and ARL, Halifax.

This work, when originally proposed in April 1984 was to have been completed by March 31, 1985 but late issue of the contract meant that work carried over into fiscal 1986.

Separate funding was to be sought for part two "Experimental Test Program on Coal and Oil Shale". Soon after issue of the contract for part one NRC ceased funding energy work and another partner to Provincial Holdings had to be found to cover the cost of the second part. An unsolicited proposal AS/84/24-P, dated February 1985, "Pyrolysis of Eastern Canadian Coals and Oil Shale in a Circulating Fluidized Bed Reactor Part 2 - Experimental

Program" was made to DSS with this in mind (UP-12-241). That proposal could not be assessed by CANMET without a report on part one. The report given here has been prepared for that purpose and it will be sent to CANMET in support of the unsolicited proposal. Note that a final report AS/86/5, May 23, 1985, had already been sent to S.G. Whiteway of ARL to complete our commitment to NRC.

## 2.0 PROCESS CONCEPT

RPC had proposed to construct and operate a fluidized bed pyrolysis unit with cyclic feed of steam and air to study the co-pyrolysis of Minto coal and oil shale from Albert County with subsequent char combustion in the same reactor. The purposes was to develop conversion technology for the indigenous coal and oil shale of New Brunswick and, at the same time, to deal with the sulphur problem attendant on coal use.

In this novel processing scheme the completion of the pyrolysis process will be followed by the initiation of co-combustion (Figure 1). Pyrolysis of a mixture of coal and oil shale will be carried out in one cycle to produce gaseous and liquid hydrocarbons and the char. In the following cycle, the product char will be combusted to raise steam. Part of the heat generated by the combustion cycle will be retained by the hot circulating solids which will be used in the subsequent pyrolysis cycle.

A concept somewhat similar to the present one has been investigated by Longwell et. al at MIT (1). However, twin fluidized bed reactors were used instead of one circulating fluidized bed as in the RPC's process. RPC's decision to use a circulating fluidized bed is based on its inherent advantages over the bubbling bed reactor. The following advantages have been reported (2,3).

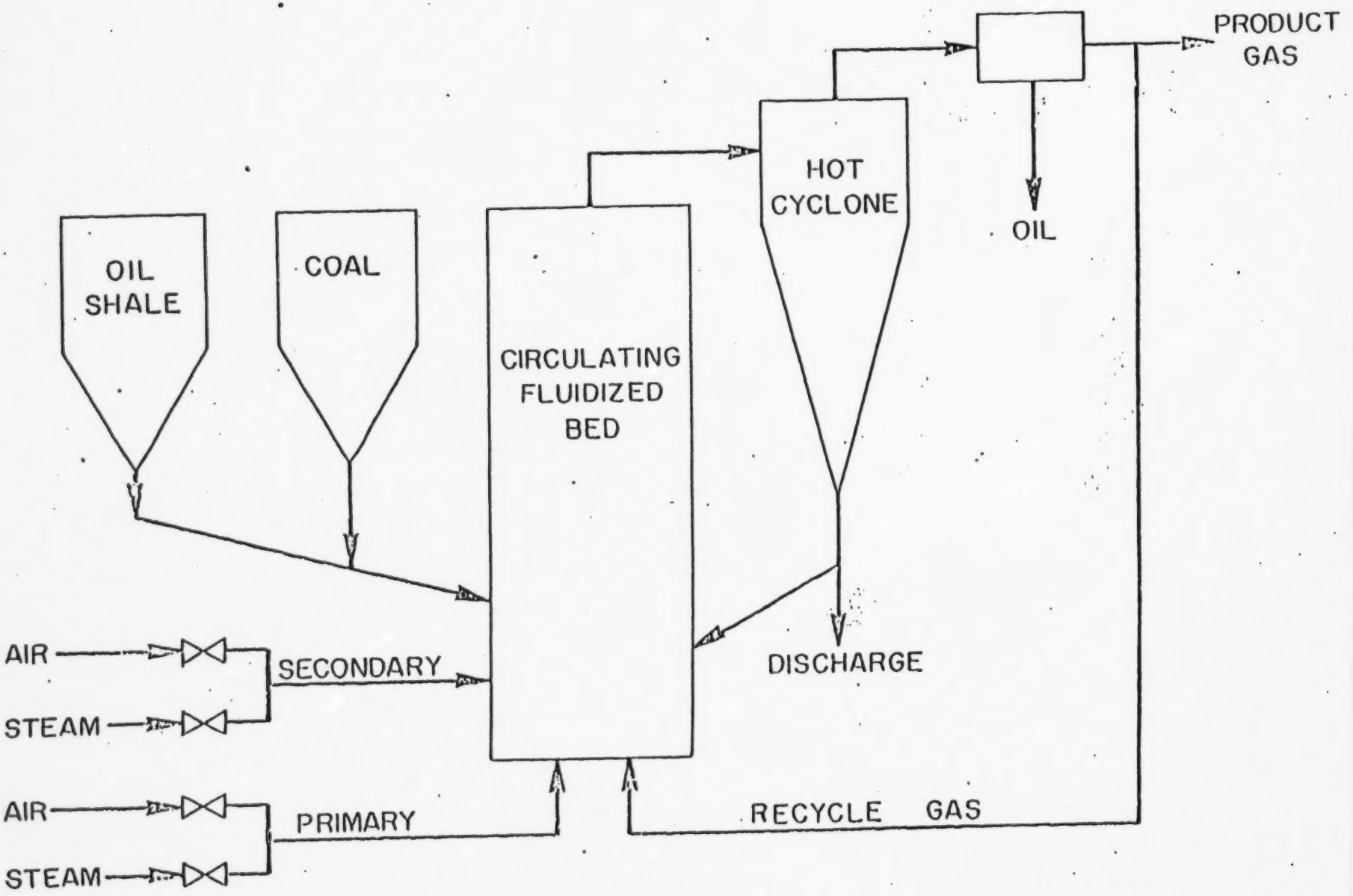


FIGURE 1: Experimental set-up for the Pyrolysis of Eastern Canadian Coals and Oil Shales

a. Capable of handling agglomerating and swelling material. New Drunswick higher sulphur coal has a high free index indicating caking tendencies. Fast fluid bed is expected to deal with this problem.

b. Almost instantaneous heating of incoming cold feed to reactor temperature. In pyrolysis this will result in a fast heat-up rate, which in turn, will improve the liquid yield.

c. Excellent gas-solid contacting which results in an efficient utilization of sorbent and emission clean-up.

d. Ease of solid feeding into the reactor.

The RPC's process is expected to have the following advantages:

a. Complete utilization of mixture of high sulphur coal and oil shale in one reactor which will result in lower capital cost.

b. Production of a pyrolytic gas free of $H_2S$. Liquid production will be maximized.

c. All the sulphur will be retained by the solid inorganic residue which will contain little recoverable energy.

## 3.0 WORK COMPLETED

It had originally been planned to modify the 6-inch fluidized bed reactor used for earlier work on coal and oil shale combustion but it soon became evident that the state of the equipment and the requirements of the planned experimental program warranted the design and construction of an entirely new unit which would be refractory lined and could withstand the harsh experimental condition to which the equipment would be subjected. It had to be refractory lined both for adequate insulation and to be able to withstand the severe erosion which is likely from abrasive circulating solid particles. Consequently, there was a shift in the planned program with the approval of the scientific authority so that the emphasis was on construction of the unit. Commissioning was to take place in part two for which funding was being requested through an unsolicited proposal.

A description of the test fairly constructed follows, and then that of the cold tests undertaken to establish that effective circulation of the material could take place.

## 3.1 TEST FACILITY

The circulating fluidized bed is shown schematically in Figure 2 and an overall view is shown in Photograph 1. It consists of a fast fluid bed reactor, a cylindrical stand pipe, two hot cyclones in series, gas cooling/liquid condensing system, gas and solid recycling network and solid feeding recycling back to the system. The description of each of the components of the system are given below. Design drawings are given in appendix A (diagrams A 1 to A10).

### 3.1.1 FAST BED REACTOR

The reactor is cylindrical in shape with a total height of 5.8 m (19/) and has an inside and outside diameter of 12.7 cm (5") and 50.8 cm (20") respectively. The wall consists of three layers of material: an inner layer made up of high temperature refractory, a middle layer of castable refractory insulation, and carbon steel shell. The thickness of the high temperature refractory and insulation are 5 cm and 13 cm respectively. A burner is provided at the bottom of the reactor for start-up.

### 3.1.2 STAND PIPE

The stand pipe is used for storage and recirculation of solids received from the primary cyclone. It is 2.7 m (9') high and has an inside and outside diameter of 22.8 cm (9") and 61 cm (24") respectively. Like the fast bed reactor, the outer shell is made of carbon steel and with a similar refractory lining. Provisions are made for the measurement of axial and radial

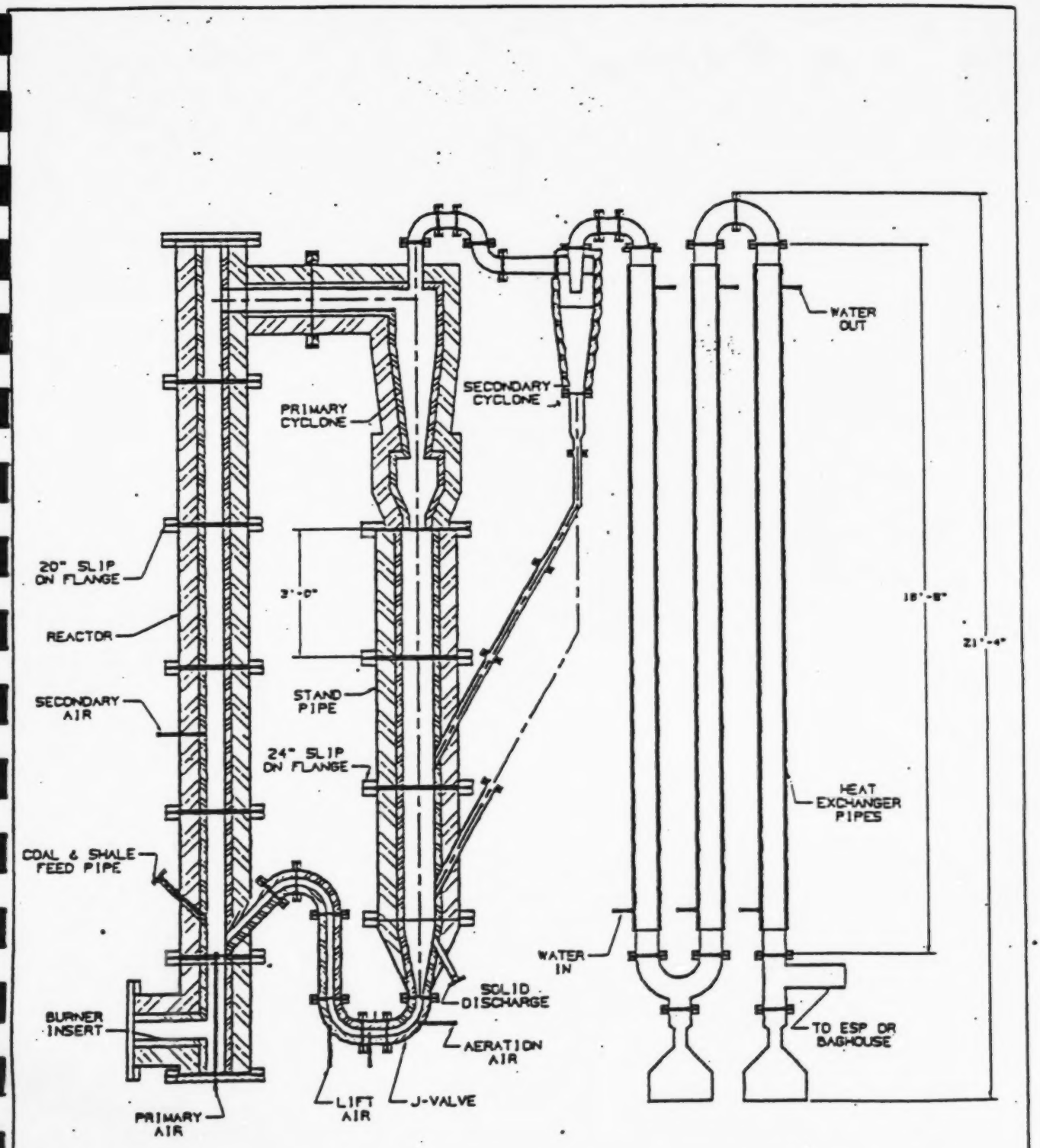


Figure 2: SCHEMATIC OF RPC 5" RECIRCULATING FLUIDIZED BED REACTOR

temperature of the standpipe. Discharge and sampling ports have been installed along the height of the standpipe.

Since no heat is generated during the pyrolysis cycle, the standpidpe has been designed to hold a sufficient amount of hot solids, heated during the combustion cycle, which helps to extend the duration of the pyrolysis cycle. If necessary, excess heat from the combustion cycle will be removed by water cooling coils. The hot solids are circulated and are transferred to the reactor by a non-mechanical J-valve.

### 3.1.3 CYCLONES

The primary cyclone which is 12" in diameter, See Photograph 2, will receive a considerable amount of hot solid, therefore it is refractory lined in a similar manner to the reactor and the standpipe. The secondary cyclone is 10" stainless steel.

The combined efficiency of the two cyclones are more than 99.5%.

### 3.1.4 GAS COOLING AND LIQUID CONDENSING SYSTEM

Off-gases from the secondary cyclone undergoes cooling and/or condensation depending on the operating mode of the reactor. During combustion the gases are cooled, metered and vented to the atmosphere. During pyrolysis, cooling of gases is accompanied by condensation of the oil mist which is collected at the bottom of the condenser. Since the gases still contain considerable amounts of oil, it is further cleaned by an electrostatic precipitator. Part of the cleaned gas is recycled to the reactor and to the

J-valve as the pneumatic transport gas. The remaining cleaned gas is sampled, metered and vented to the atmosphere.

First stage cooling and/or condensation is carried out in three water cooled one pass heat exchanger/condenser series. These heat exchangers consist of two concentric pipes where coolant flows through the annulus. The nominal diameter of the inner and outer pipe are 6" (Schedule 40) and 8" (Schedule 60) respectively. The height of each exchanger is 5.2 m (17"). The electrostatic precipitator is a five stage collection of cells.

3.1.5 SOLID AND GAS RECYCLING NETWORK

During the pyrolysis cycle, the clean gases are recycled to the reactor and the J-valve as fluidizing and pneumatic transport gases. (During combustion cycle air is used). Essentially all the air streams used during the combustion cycle are to be replaced by the recycle gas during the pyrolysis cycle.

Circulation of solids from the standpipe to the reactor is achieved by the non-mechanical J-valve, See Photograph 3. Air/gas is supplied at two points - one at the base of the standpipe, the other as the lifting media in the vertical section of the J-valve. The J-valve is made of 2", 40 Schedule SS pipe.

### 3.1.6 SOLID FEEDING

Coal and oil shale are to be fed into the bed by individual screw feeders equipped with variable speed motors, see Photograph 4. Each feeder is placed on four load cells which provided an account of the solid feed rate. The solids discharged by the screw feeders will be fed to the reactor either pneumatically and/or by gravity.

### 3.1.7 GAS SAMPLING SYSTEM

On-line gas analyzers, see Photograph 1, (hydrocarbon, CO, $CO_2$, $O_2$) will be used to monitor the composition of off-gases and the recyle gases. Sample gases will be continuously withdrawn from the flue gas/recycle gas and further cleaned by a series of filters prior to entering the analyzers.

## 3.2 COLD TESTS

The primary objective of the cold tests was to assess the performance of the J-valve which is one of the critical areas in the design of criculating fluidized bed reactors.

Sand with size fractions - 35 to +80 mesh (177 to 420 microm) was loaded into the standpipe to give a bed height of approximately 7½ ft measured from the bottom. The aeration and lift air was supplied by a compressor at a pressure of 20 psi.

The sand transferred by the J-valve was collected at the bottom of the reactor and weighed. A number of runs have been made varying the rate of aeration and lift air to evaluate the amount, characteristics and control of the solid flow. The J-valve was intermitently operated for approximately six hours. The duration of each run which which was used to calibrate the J-valve did not exceed ten minutes.

A maximum flow rate of 1700 kg/hr was achieved with approximate 70% of the air supplied as the aeration air and the remaining 30% as lift air. When the amount of aeration and lift air was reversed, the solid flow rate dropped to approximately 800 kg/hr which was the minimum flow rate achieved. Other intermediate flow rates were achieved by adjusting the amount of aeration and lift air.

The flow of solids in the J-valve was not continuous, rather, it was in the form of slugs which are only a fraction of a second apart and at an irregular interval. It is not expected that a pulsating flow will affect the performance of the reactor and to all practical purposes the flow will be considered to be Continuous.

The J-valve was constructed from 2 inch, 40 schedule, (0.154" 316SS) stainless steel pipe, which unlike other major components was not refractory lined. As it is probable that there will be erosion of metal at the bends from circulating abrasive particles plans are underway to replace it with a refractory lined unit of similar design.

## 4.0 PROJECT FINANCES

Funds allocated to the project were $85,127.40. NRC through DSS contributed $43,097.40 and the balance has been provided by the N.B. Government by Provincial Holdings Limited.

The project exceeded budget by approximately $100,000 because contrary to the original plan, a new refractory lined reactor was constructed rather than modifying the existing stainless steel fluidized bed unit. The extra cost is being absorbed by RPC.

## 5.0 CONCLUSIONS

The construction of the entire system is essentially complete and it is ready to be operated under normal operating conditions. The non-mechanical J-valve, the performance of which has not been widely reported in the literature, has functioned satisfactorily according to its design specifications. The equipment will be used immediately for the DSS contract "Modification of Integrated Oil Shale Retort System (DSS file No 06SQ.23440-5-9028).

The decision to construct a refractory lined reactor is justified (despite the extra cost) in the light of the abrasive, erosive nature of the circulating solids.

# REFERENCES

1. Longwell, J.P.; Howard, J.B., Peters, W.A.; and Yeboah, Y.D.; Coal Pyrolysis by Hot Dolomitic Solids from a Fluidized Bed Combustor", paper presented at the Workshop on Application of Pyrolysis Technology to the Utilization of Eastern Canadian Coals, Atlantic Research Laboratory Technical Report 30, NRCC 19903 (1982).

2. Yerushalmi, J.; Cankurt, N.T.; "High Velocity Fluid Beds", CHEMTEDCH, Vol. 8, Sept. 1978, 564-572.

3. Yip, H.H.; Engstrom, F.; "Operating Experience of Commercial Scale Pyroflow Circulating Fluidized Bed Combustion Boilers", The 7th International Conference on Fluidized Bed Combustion, Philadelphia, Pennsylvania, Oct. 25-27, 1982.

# AUTHORIZATION

DEPARTMENT: Applied Sciences

PROJECT TITLE: Pyrolysis of Eastern Canadian Coals and Oil Shales in a circulating fluidized bed reactor

PROJECT NUMBER: PROPOSAL NUMBER

REPORT AS/85/16

Prepared By: P. Salib & S. Barua

Reviewed By: P. Salib

Approved By: D. Abbott

Date: August 16, 1985

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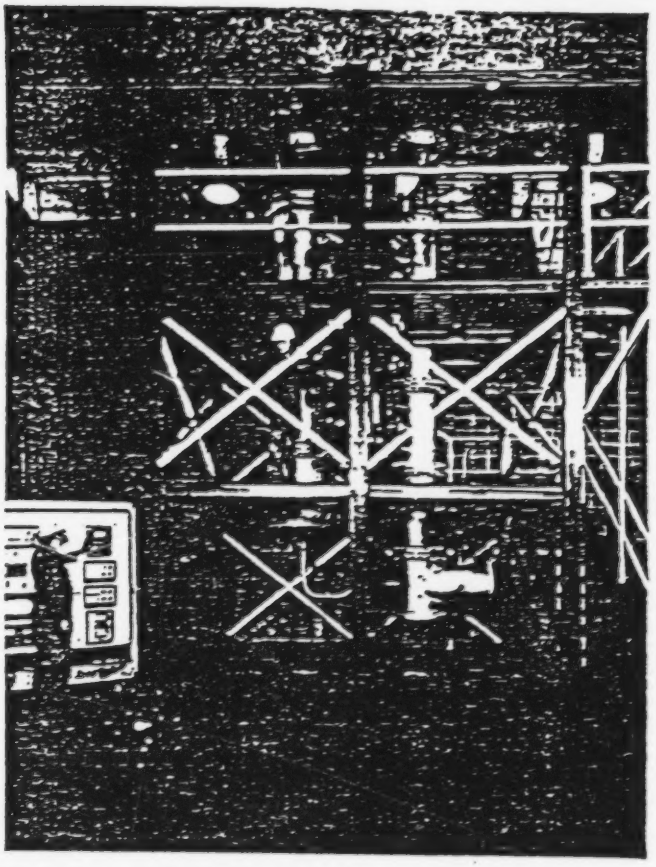
# PHOTOGRAPHS

1. Overall view of the Circulatory Fluidized Bed Reactor.

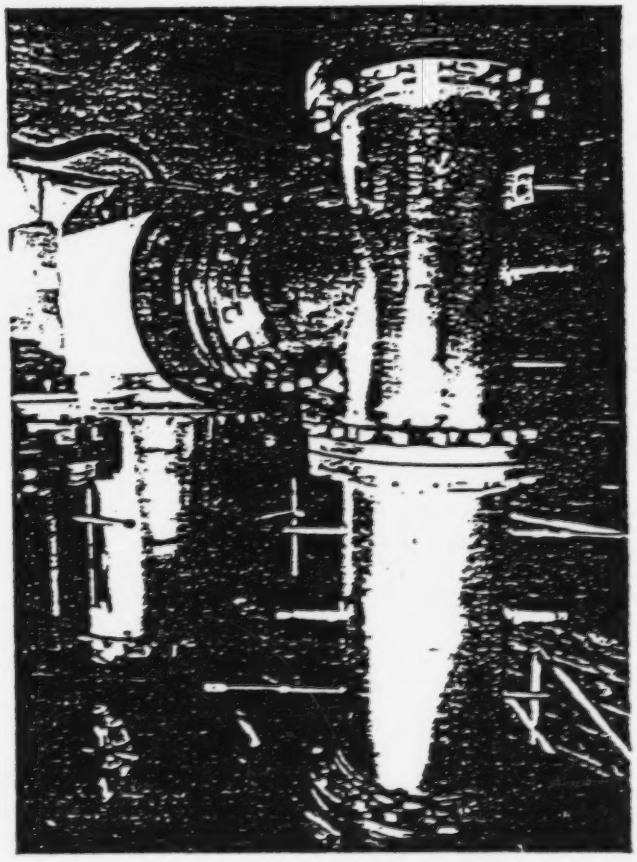
2. View of the Primary Cyclone and the top section of the reactor.

3. Non-mechanaical J-valve for cirulation of solids from standpipe to reactor. (External insulation not in place).

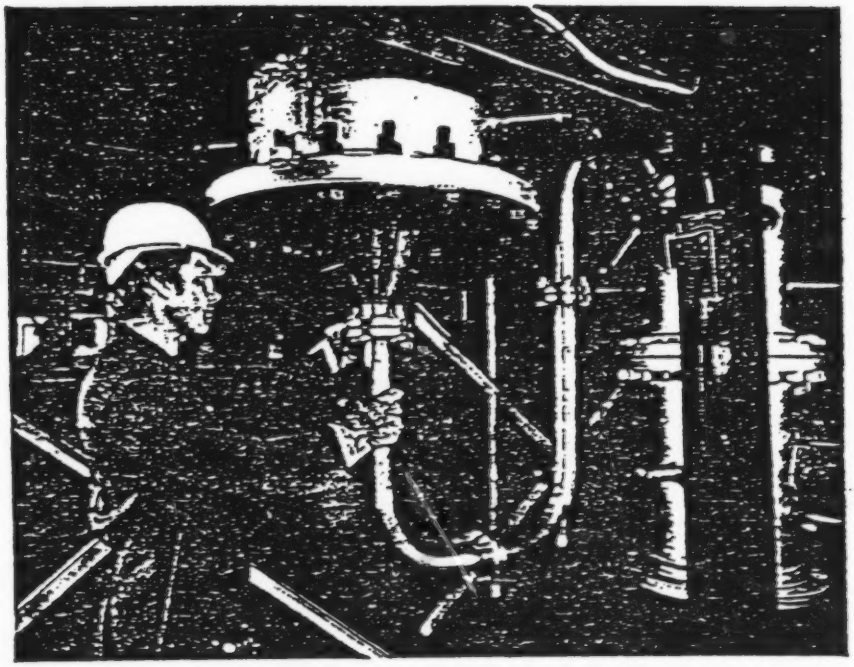
4. Raw material hoppers and feeding system.



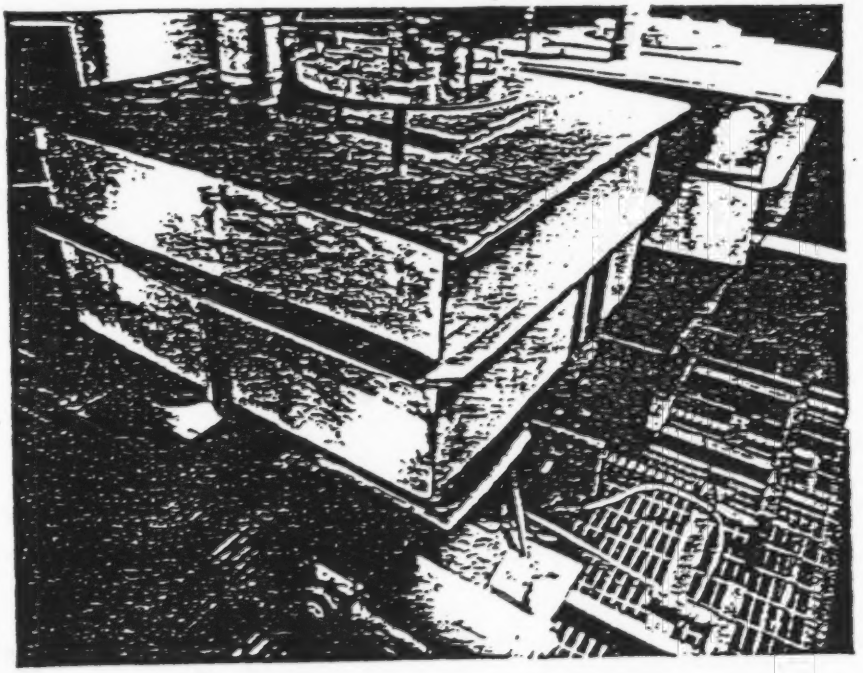
Photograph 1: Overall view of the Circulating Fluidized Bed Reactor



Photograph 2: View of the Primary Cyclone and the top section of the reactor.



Photograph 3: Non-mechanical J-valve for Circulation of solids from standpipe to reactor. (External insulation not in place)



Photograph 4: Raw material hoppers and feeding system

# APPENDIX 'A'

## DESIGN DRAWINGS

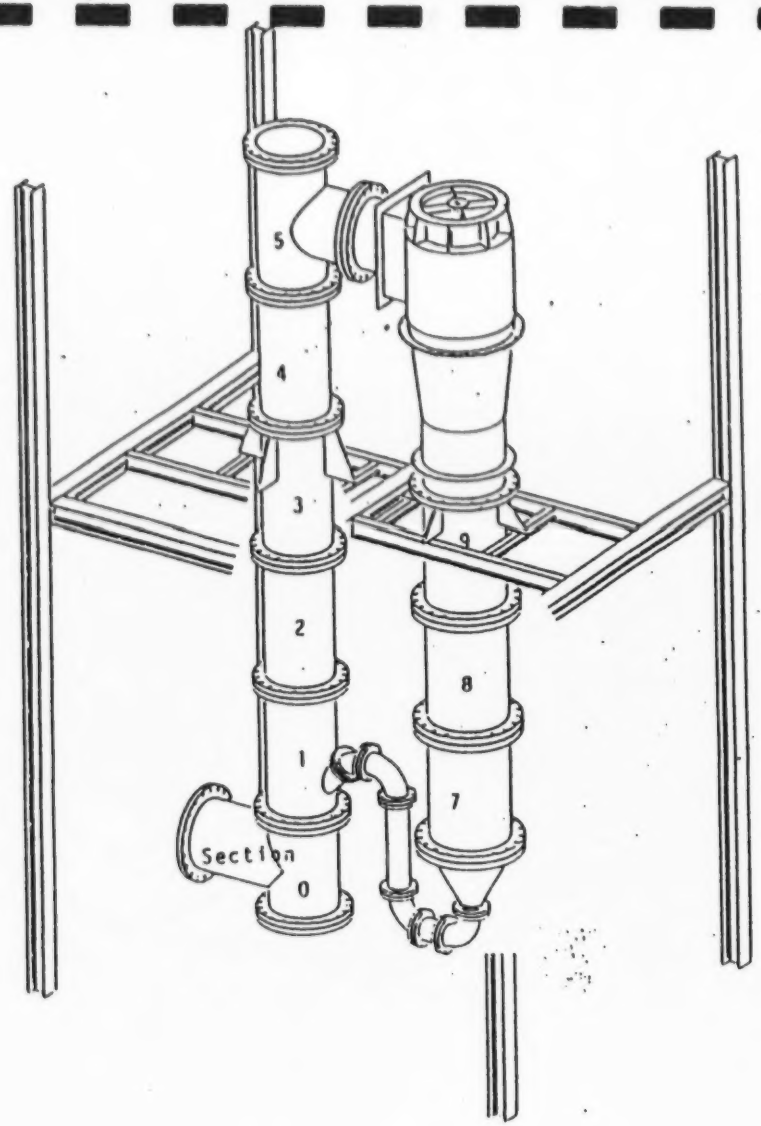



FIGURE A1: A 3-D Assembly of the Circulating Fluidized Bed Reactor showing the different sections and the Primary Cyclone

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DESCRIPTION 3-D SCHEMA... STANDPIPE

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|---|---|---|---|---|
| | | | | |

DRN: M. MENARD DATE: 10/04/1995

CK'D: DATE:

SCALE .03251:.0

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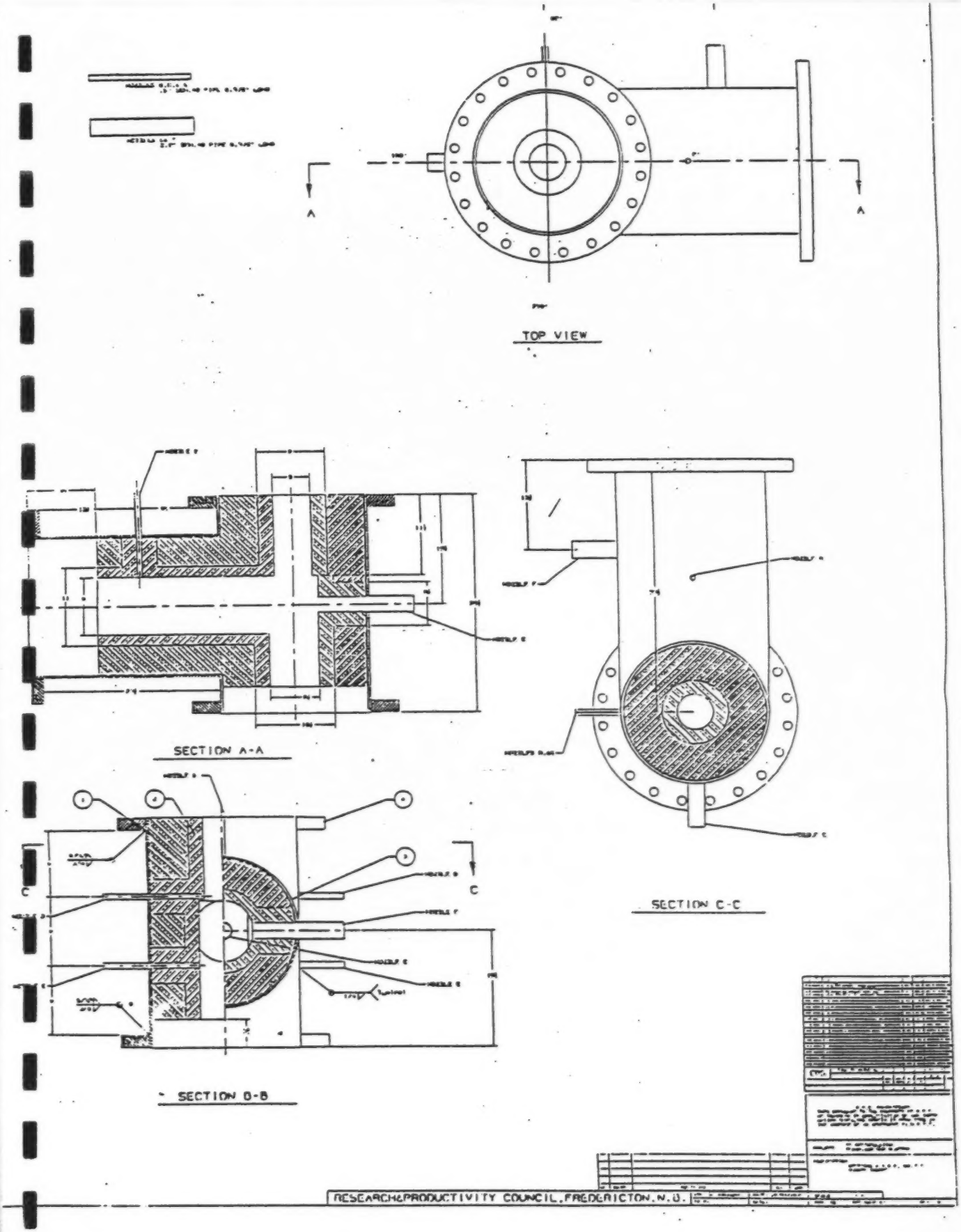
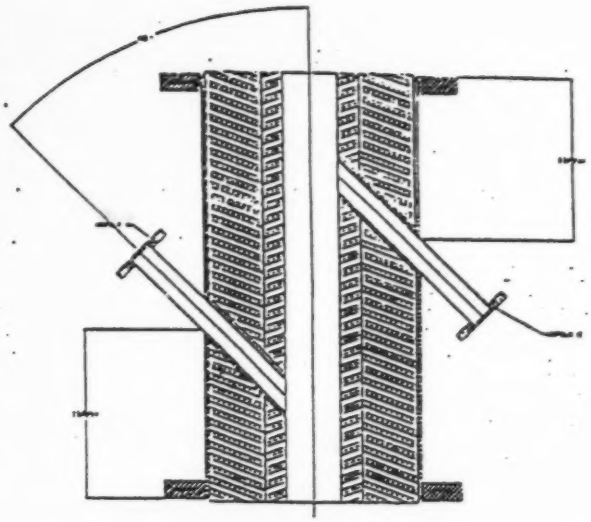
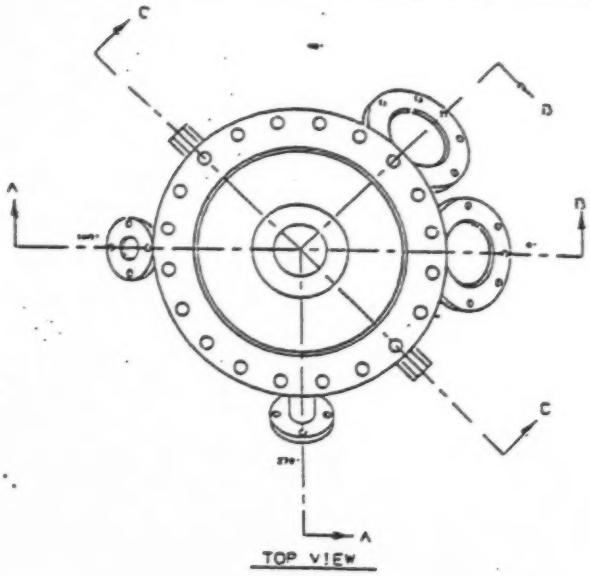


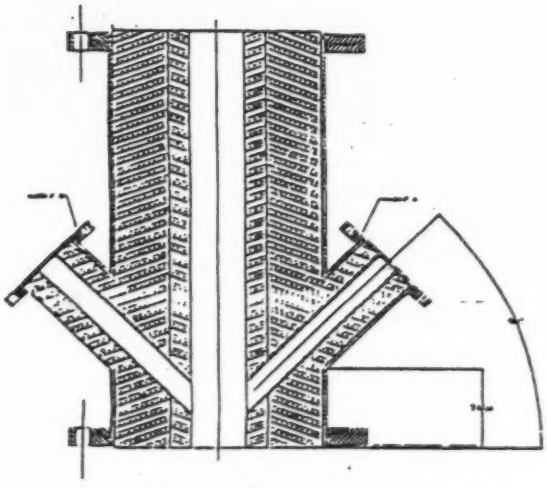

FIGURE A2: Design drawings of Section "B"



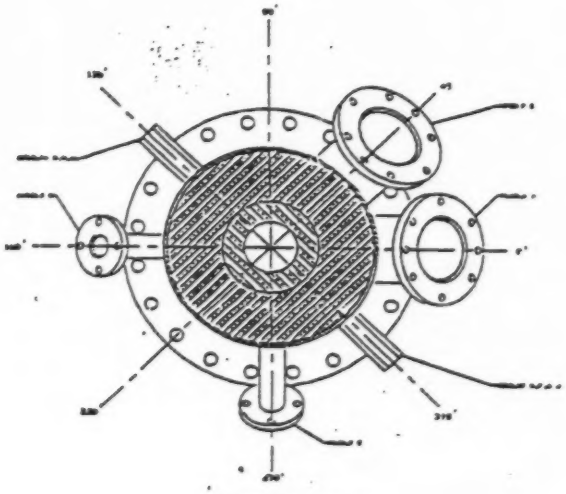
SECTION A-A



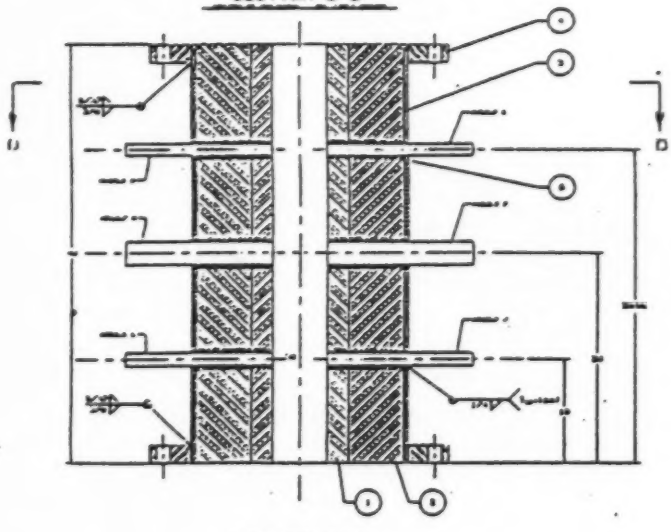
TOP VIEW



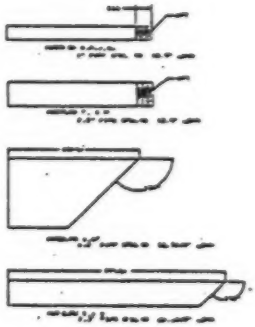
SECTION B-B



SECTION D-D



SECTION C-C



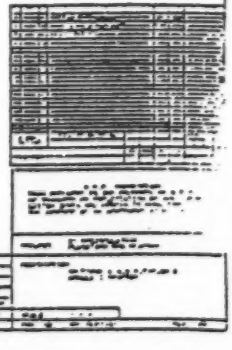


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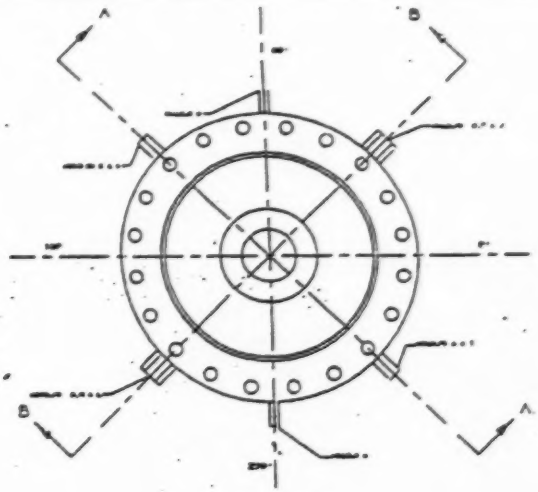
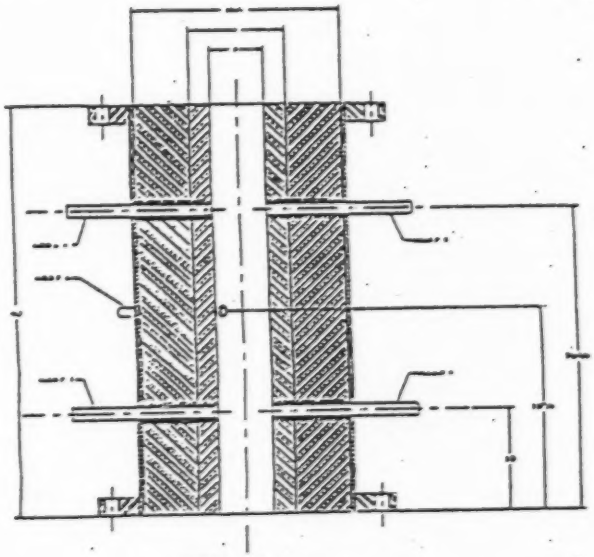
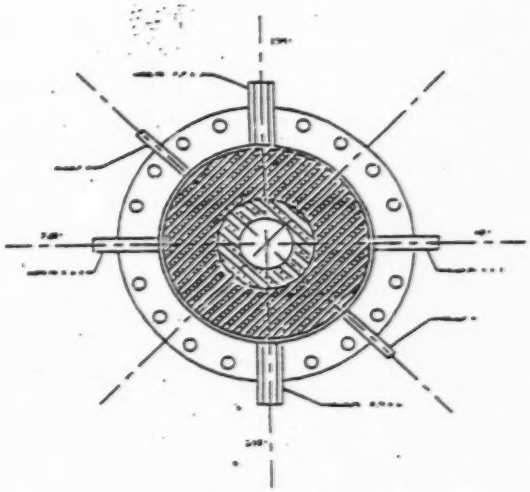
FIGURE A3: Design drawings of Section "1"



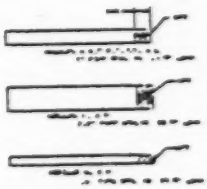

TOP VIEW

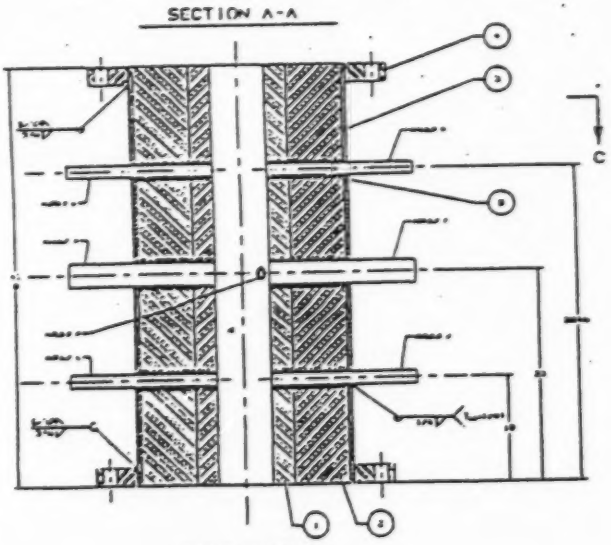


SECTION A-A



SECTION C-C






SECTION B-B

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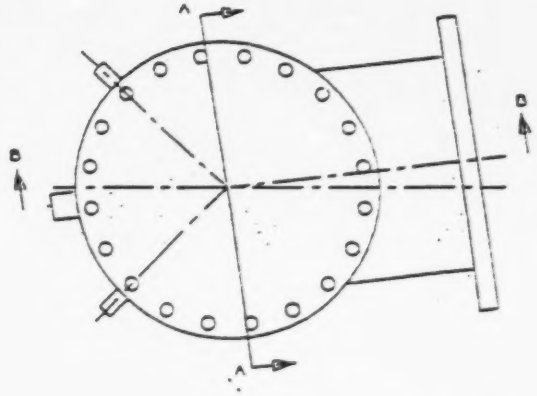
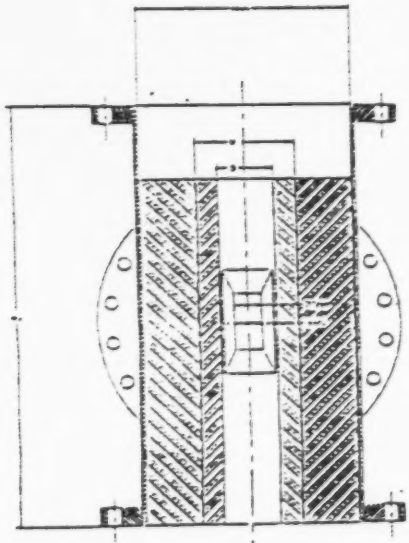
FIGURE A4: Design drawings of Section 2

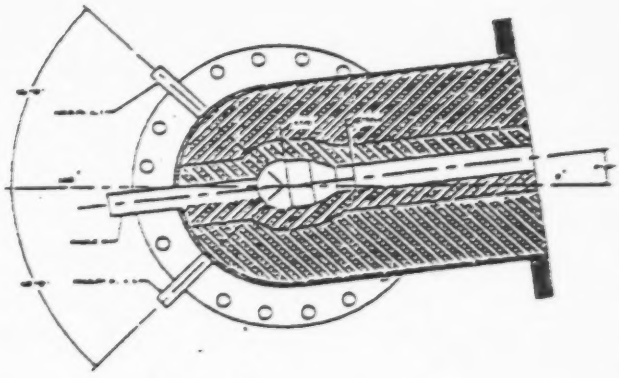
SECTION B-B
SECTION A-A



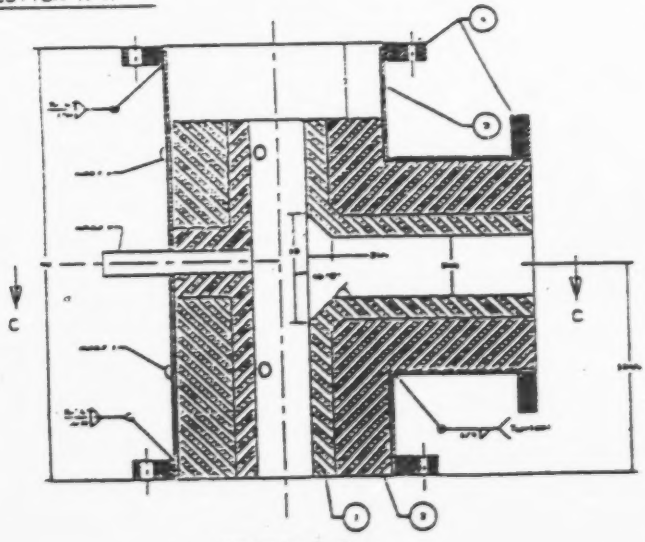

TOP VIEW



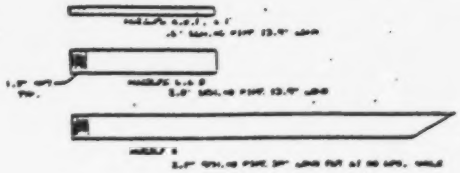
SECTION A-A

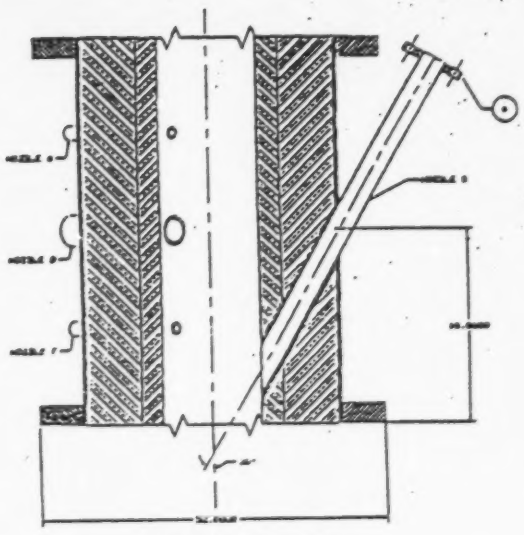


SECTION C-C

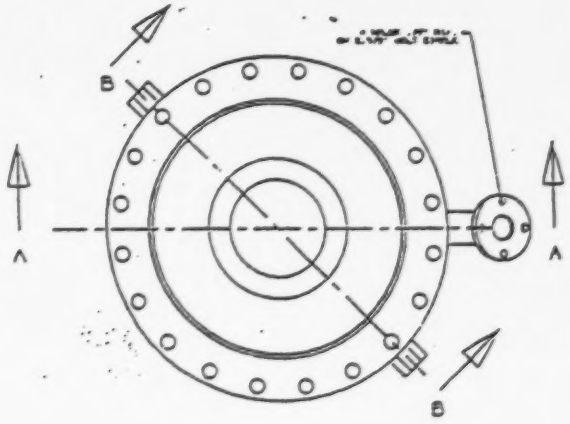


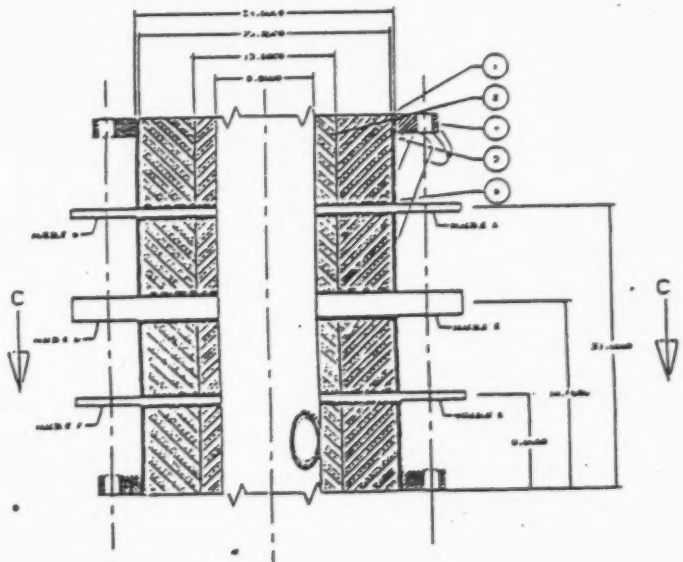

SECTION B-B



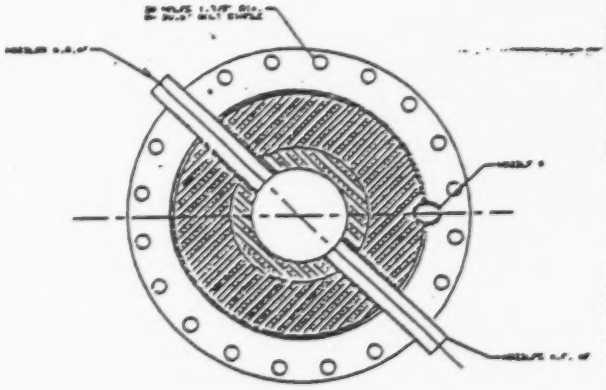


SECTION A-A




SECTION B-B



SECTION C-C

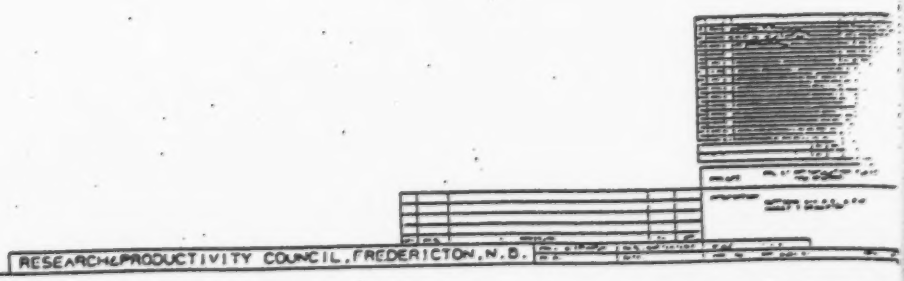


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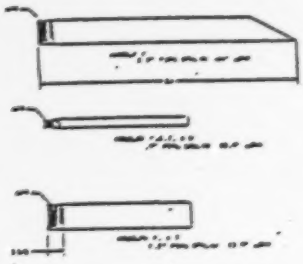
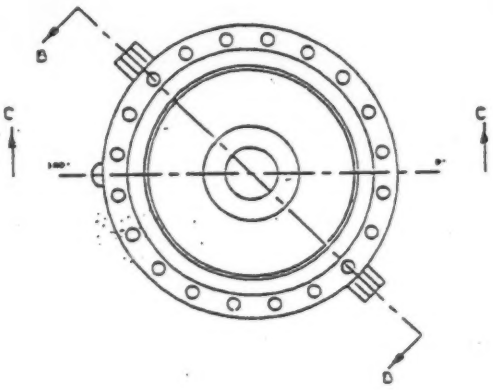
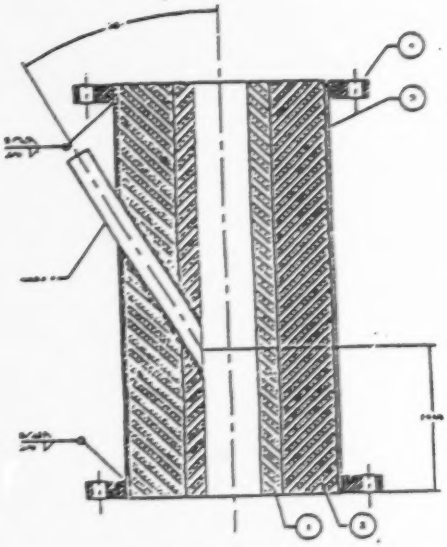
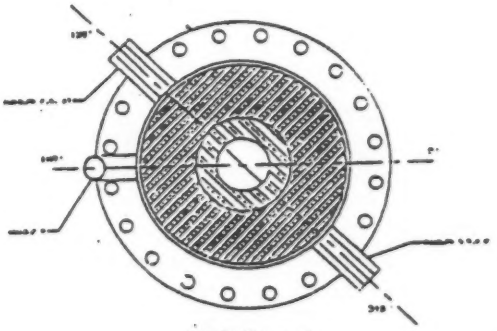
FIGURE A7: Design drawings of Section 7



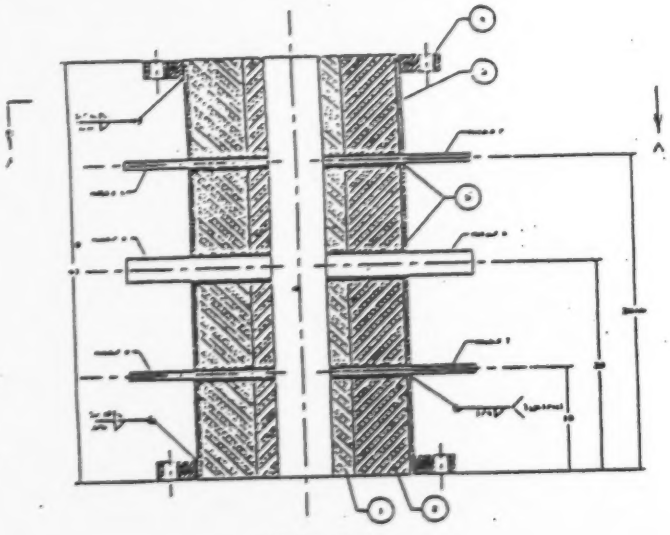


SECTION C-C



SECTION A-A



SECTION B-B

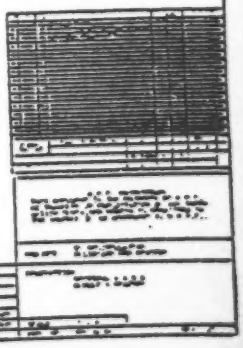
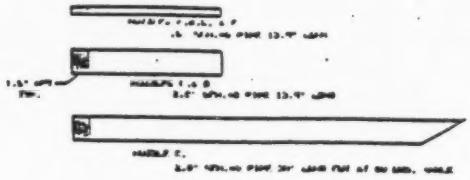
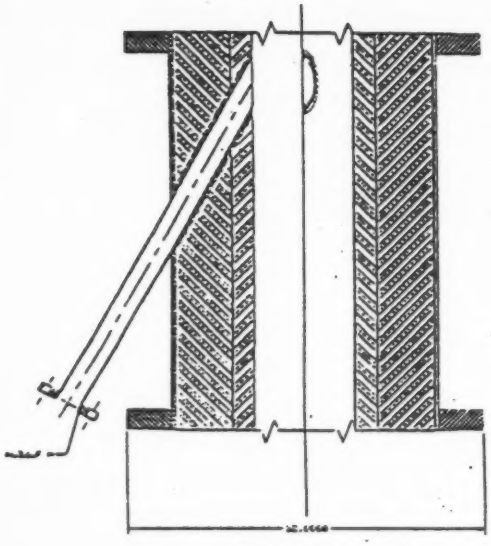
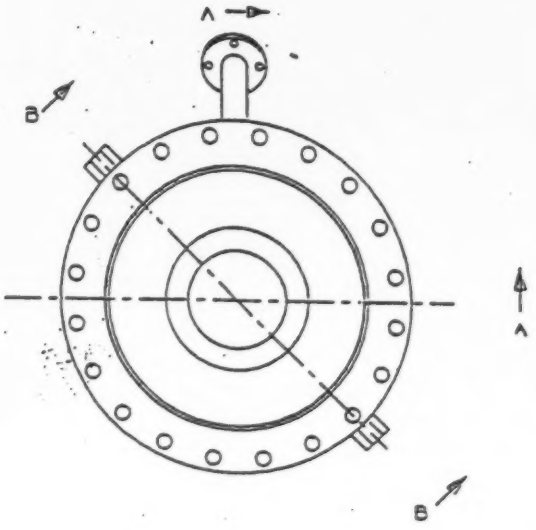


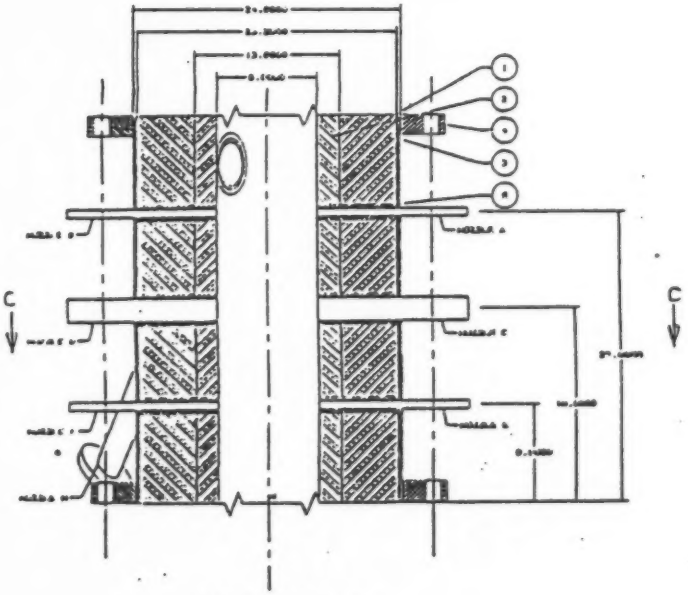

FIGURE A8: Design drawings of Section 8





SECTION A-A

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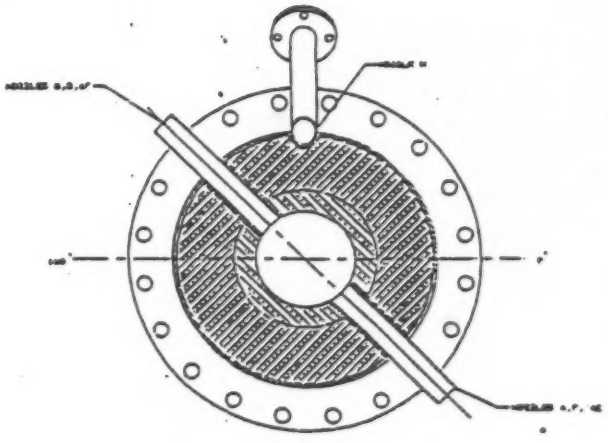
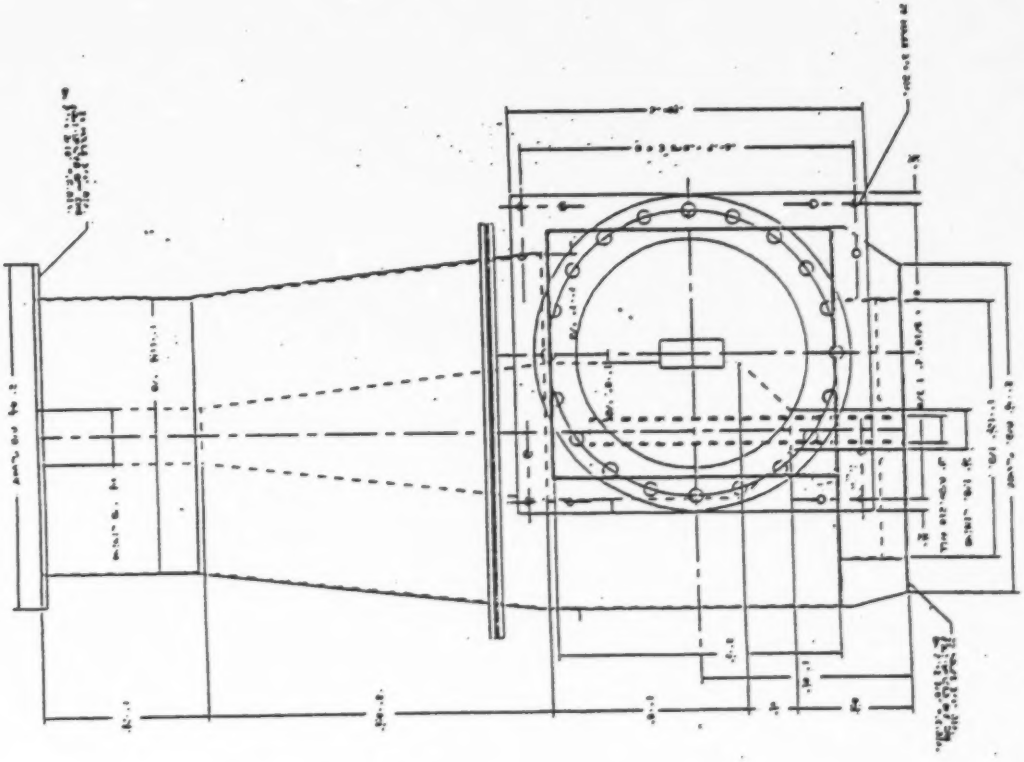
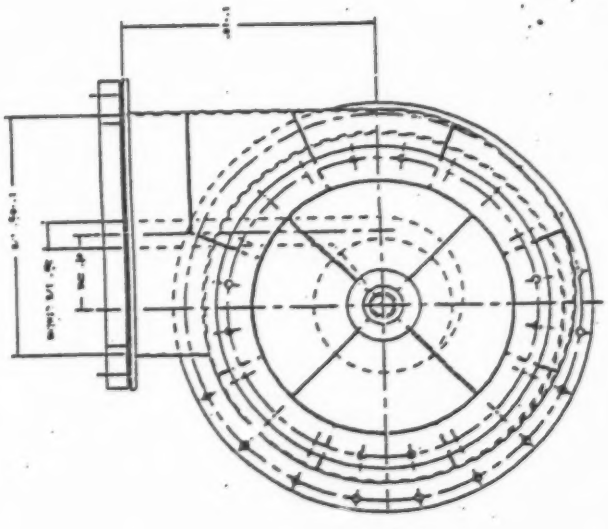


SECTION C-C





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